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EFFECT OF VOIDS ON MECHANICAL PROPERTIES OF GRAPHITE FIBER COMPOSITES

December 1970

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SYSTEMS DIVISION
Lowell Industrial Park
Lowell, Massachusetts 01851

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13. ABSTRACT

The results of an investigation of the effect of voids on the mechanical properties of Thornel 50/Epoxy and Modmor II/5206 Epoxy are discussed and presented. Unidirectional, as well as quasi-isotropic, laminates with symmetrical and nonsymmetrical ply-stacking sequences were fabricated with high and low porosity and subsequently subjected to detailed nondestructive and destructive testing. Longitudinal and transverse flexure and tension, short beam shear, and torsion rod experiments were completed on the two composite systems at 75 and 250 F. Ultrasonic compression and shear wave velocities were measured at discrete locations on a specimen-by-specimen basis, and the observations correlated with observed mechanical properties.

In addition, an in-depth metallographic characterization of voids was completed on Thornel 50/Epoxy and correlation established for NDT - mechanical properties and voids. These relationships were used to estimate upper and lower bounds on the strength and stiffness envelopes of the unidirectional and angle-ply composites. This permits evaluation of the degradation effects of voids on composite performance.

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Thornel 50						
Nondestructive Testing						
NDT Correlations for Composites						
MODMOR II/5206 Epoxy						
Strength and Stiffness Envelopes						

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EFFECT OF VOIDS ON MECHANICAL PROPERTIES
OF
GRAPHITE FIBER COMPOSITES

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FOREWORD

The research reported herein was performed by Avco Systems Division, Lowell Massachusetts, Dr. E. M. Lenoe, Principal Investigator, under Department of the Navy Contract N00019-70-C-0242, entitled "Effect of Voids on Mechanical Properties of Graphite Fiber Composites. This program was administered through the Naval Air Systems Command under the cognizance and direction of Mr. M. Stander. The inclusive dates of this work effort were May 1970 to December 1970.

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SECTION I

1.0 INTRODUCTION AND SUMMARY

The work reported herein represents the results obtained in the third yearly phase of "The Effects of Voids on Graphite Fiber/Epoxy Composites". For the convenience of the reader, the prior year objectives and results are summarized below:

1. DETERMINATION OF VOID CONTENT

   Four techniques were examined to assess their applicability in determining the void contents of graphite epoxy composite materials and the limits of precision were determined for each procedure. The four methods were:

   - Resin Burnout - heating of the graphite epoxy composite to 425°C for at least 4 hours, such that the epoxy resin burns out, yet the graphite fibers are unaffected.
   - Acid Digestion - placing the graphite epoxy in nitric acid to dissolve the epoxy resin.
   - Thermogravimetric Analysis (TGA) - heating of the graphite epoxy at a constant rate and observing a relatively flat region of the TGA due to oxidative degradation of the epoxy resin just prior to final drop-off.
   - Point Count Metallography - using photomicrographs of sample cross sections in conjunction with a fine grid to count (calculate) void area versus graphite epoxy area.

   Void contents were then calculated for specimens subjected to the resin burnout, acid digestion, and TGA techniques. Based upon a consideration of the effect of the accuracy and preciseness of resin weight and fiber density on the calculated void content, it was concluded that for even a 0.1% coefficient of variation in each of the fiber density, resin density, composite density, and fiber volume fraction, the variance in the void content was 2.46%. Hence, since the coefficient of variation for resin content determination from these three procedures was better than 2% and the fiber density variation can be as great as 7%, and the coefficient of variation on composite density is not less than 0.1%; it was concluded that these techniques are only adequate to determine gross void content --but obviously not able to determine void size, shape or distribution. Hence, all subsequent void content determinations were made using the point count metallography techniques.

2. THEORETICAL EFFECTS OF VOIDS

Concurrently, a review of theoretical considerations of void effects on the matrix resin and such composite materials was made in order to determine the applicability of such models and, if applicable, to use such models to determine the bounds of void effects on mechanical properties. These results are discussed in detail in Reference 1.

3. EFFECT OF VOIDS ON THE MECHANICAL PROPERTIES OF THORNEL 50/EPOXY COMPOSITES AND MODMOR II/5206 COMPOSITES

Composite laminates (52% fiber volume fraction) were fabricated using typical and some deliberate processing procedures to introduce a range of void contents estimated from 1/2 to 11%. Subsequently, longitudinal and transverse flexural strengths and moduli and shear strengths were measured. The range in measured properties for these laminates, containing an estimated 1/2 to 11% range in void content, were as indicated below:

Property	Range
Longitudinal Flexural Strength	127 to 75 ksi
Longitudinal Flexural Modulus	25 to 20 ksi
Transverse Flexural Strength	6 to 1 ksi
Transverse Flexural Modulus	940 to 290 ksi
Horizontal Shear Strength	6 to 4.2 ksi

Hence, the work effort described herein consisted of the following tasks:

1. Determination of void contents by point count metallography techniques for the Thornel 50/epoxy longitudinal and transverse flexural and shear test specimens.

2. Fabrication and test of "resin only" specimens, with a range of void contents to determine the effect of voids on mechanical properties.

3. Fabrication and test of Thornel 50/Epoxy and Modmor II/5206 specimens, with a range of void contents to determine the effect of voids on mechanical properties of these composites.

SECTION II

2.0 VOIDS: THEIR SOURCE, MEANING, AND CONTROL

2.1 Source of Voids

As used in the context of this report, a void is a gas bubble which has been trapped within the cured fiber-reinforced resin composite material. Both the material constituents and the fabrication processes are contributory sources of voids. The constituents are the resin and the graphite fiber yarn (or tow) bundles; the processes are pre-impregnation of the fiber bundles, laminate layup, and curing of the resultant part. When the cure is advanced too quickly, voids are formed from the vapor of the resin or solvent. Furthermore, some of the resin ingredients may be volatilized if the temperature steps of the cure cycle exceed the rate at which the cross-linking occurs during polymerization; for example, ERLA 0300, a diepoxide component will vaporize at initial temperatures in excess of 180°F unless the reaction with the hardener has proceeded at that temperature sufficiently to tie up nearly 100% of the diepoxide. In the hardeners, anhydrides, like methyl nadic anhydride, have an initial limitation of 250°F for the same reason. However, since volatilization occurs when the resin is in a low viscosity state, the gas bubbles do, somewhat, migrate through the resin bundles and, unless they are impeded by the fiber bundles,can possibly escape.

At the present time, the majority of graphite epoxy broadgoods is processed into laminates via hand layup techniques; the plys must be cut, trimmed, and oriented and finally stacked to form a prepreg laminate. This assembly is then consolidated and cured at elevated temperatures. This discussion should lead the reader to the fact that the particular fabrication process, with all its steps, should result in a certain type and range of voids. For the Thornel 50/epoxy laminates fabricated with typical procedures, the resulting void contents, determined by point count metallography, ranged from 2 to 5%.

2.2 Effect of Voids

The effect of voids in the context of this study is their influence on mechanical and structural properties. These effects must be considered from two viewpoints, namely:

1. Quality Control Effects
2. Design Allowables Effects

The distinction is necessary for a variety of reasons. First, it is imperative to note that when dealing with mechanical properties testing of high-modulus, high-strength composites, basic distinctions must be made between the use of different test methods. Flexure tests, for example, and short beam shear tests are not adequate for use as preliminary design allowables data. However, these same tests play an important role in quality control procedures. Both the transverse flexure and short beam shear tests are especially sensitive to resin quality, fiber surface treatments, porosity, and general changes in laminate quality and are, therefore, very useful as process control procedures.

Detailed results describing the effect on mechanical properties of voids on both the resin and the composites will be covered in a subsequent section, as will also be a discussion on the effects of voids on preliminary design allowables.

2.3 Control of Voids

Characterization Techniques

Meaningful control of voids requires knowledge as to the effect of porosity on the performance of the laminate. Only with reliable data on the controlling influence of voids on laminate response under the various possible stress states can estimates be made as to the allowable levels of void contents.

In addition, the methods to characterize the voids are also necessary. Previously (1)*, comparative studies were made of results obtained by several methods of void determinations. These void characterization techniques included:

- Resin Burnout
- Acid Digestion
- Thermogravimetric Methods
- Metallography

The first three techniques utilized specimens of the order of 1/4" x 1/4" x 3/4" and provided an estimate of the average void content. A more exacting means of characterizing voids is based upon a metallographic procedure referred to as a "point count nodal analysis" (2), which relies upon:

- Sectioning Specimens and Polishing
- Using Enlarged Photomicrographs
- Superimpose Small Grid
- Use Statistical Procedures to Characterize Voids
- Calculate Average and Local (location and size) Voids Contents

* Numbers in parentheses pertain to the references.

During this past year, we obtained an additional 100 photomicrographs on specimens which were previously characterized by ordinary laboratory procedures (resin burnout, acid digestion, etc.). This permitted direct comparison of the alternate techniques. Resultant data appears in Figure 1. From this illustration, it appears that the gross void content determinations considerably underestimated voids up to the 6 to 8% level. Obviously, the specimen sizes and test procedures ought to be revised to obtain improved porosity characterization by these more commercial chemical laboratory methods.

However at the present time, our conclusion is that point-count nodal analysis methods of void characterization must be resorted to in order to achieve realistic information. Assessment of the effect of voids on the mechanical behavior of composites must be based on accurate knowledge of the type, size, distribution, and amount of porosity.

Monitoring Techniques

In addition to detailed knowledge of the influence of well characterized voids on composite performance, we also need methods to monitor the presence of voids. Ideally, these methods ought to be nondestructive and easily applied. In the past, ultrasonic techniques have been widely used to survey materials quality. For a composite material, wave phenomena is markedly influenced by a number of factors. These include:

- Properties of the matrix
- Properties of the fiber
- Geometry and relative proportions of the constituents
- Amount, nature, and distribution of porosity
- Matrix/fiber interface conditions, etc.

For example, it is evident that compression wave velocities will be strongly affected by changes in fiber volume fraction. This is emphasized by Figure 2 which illustrates the change in relative ultrasonic velocity as a function of change in fiber content. Suppose, now, that we are dealing with a production operation wherein the volume fraction of fibers is controlled to within a few percent, an interesting and relevant question is: What is the relative sensitivity of ultrasonic observations to porosity changes? This matter was investigated experimentally, and results for the Thornel 50/epoxy system are shown in Figure 3. The data suggests the normalized ultrasonic measurements are indeed sensitive to the presence of voids, and this fact gives us added confidence in the possibility for qualitative use of nondestructive measurements for laminate control.

CONCLUSIONS

USING TYPICAL LAB PROCEDURES AND GROSS VOID CONTENT DETERMINATIONS, 87% OF THE TIME THE LOCAL VOID CONTENT IS UNDERESTIMATED

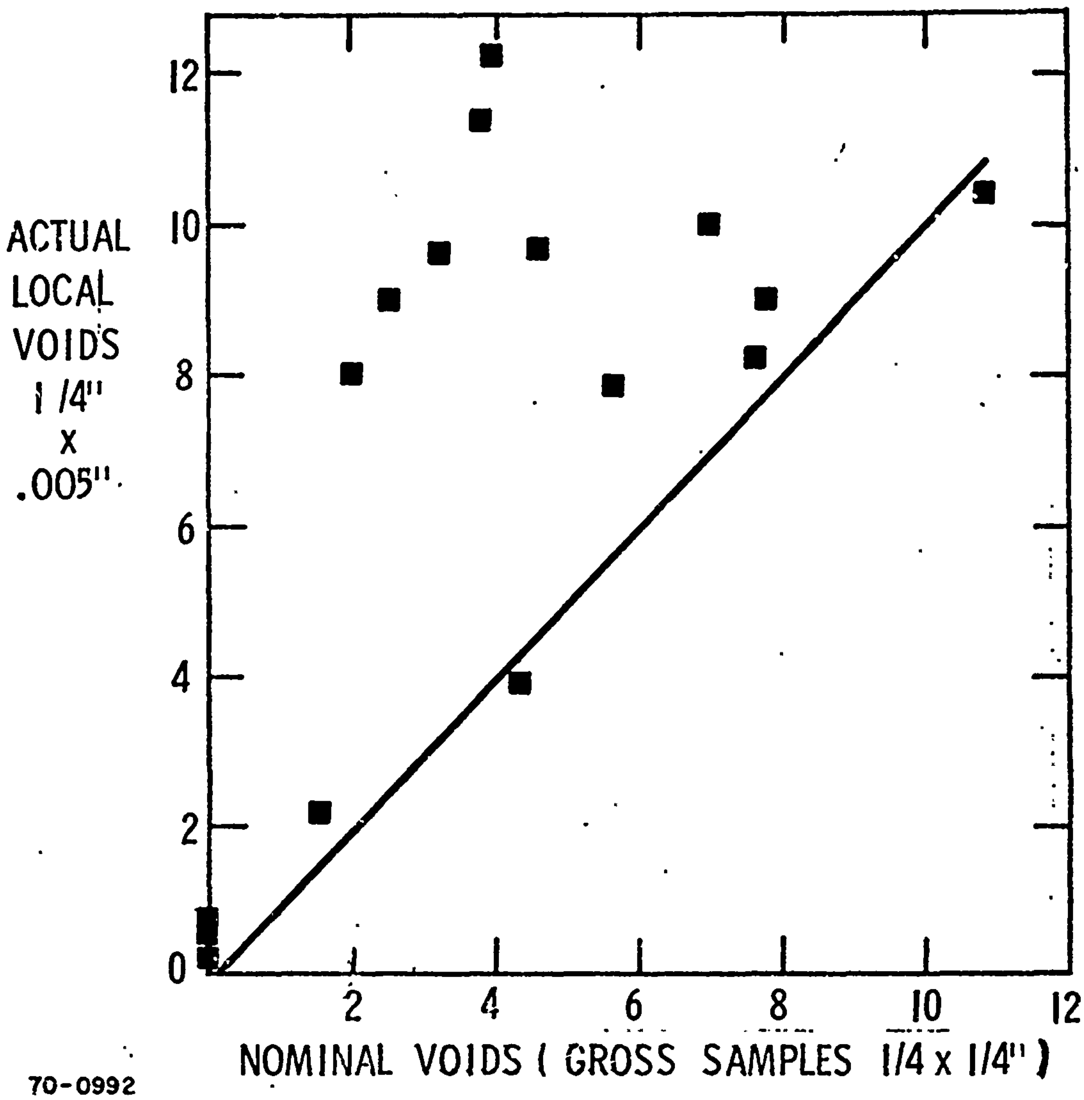

FIGURE 1 ACTUAL VOID CONTECT VS. NOMINAL VOID CONTENT

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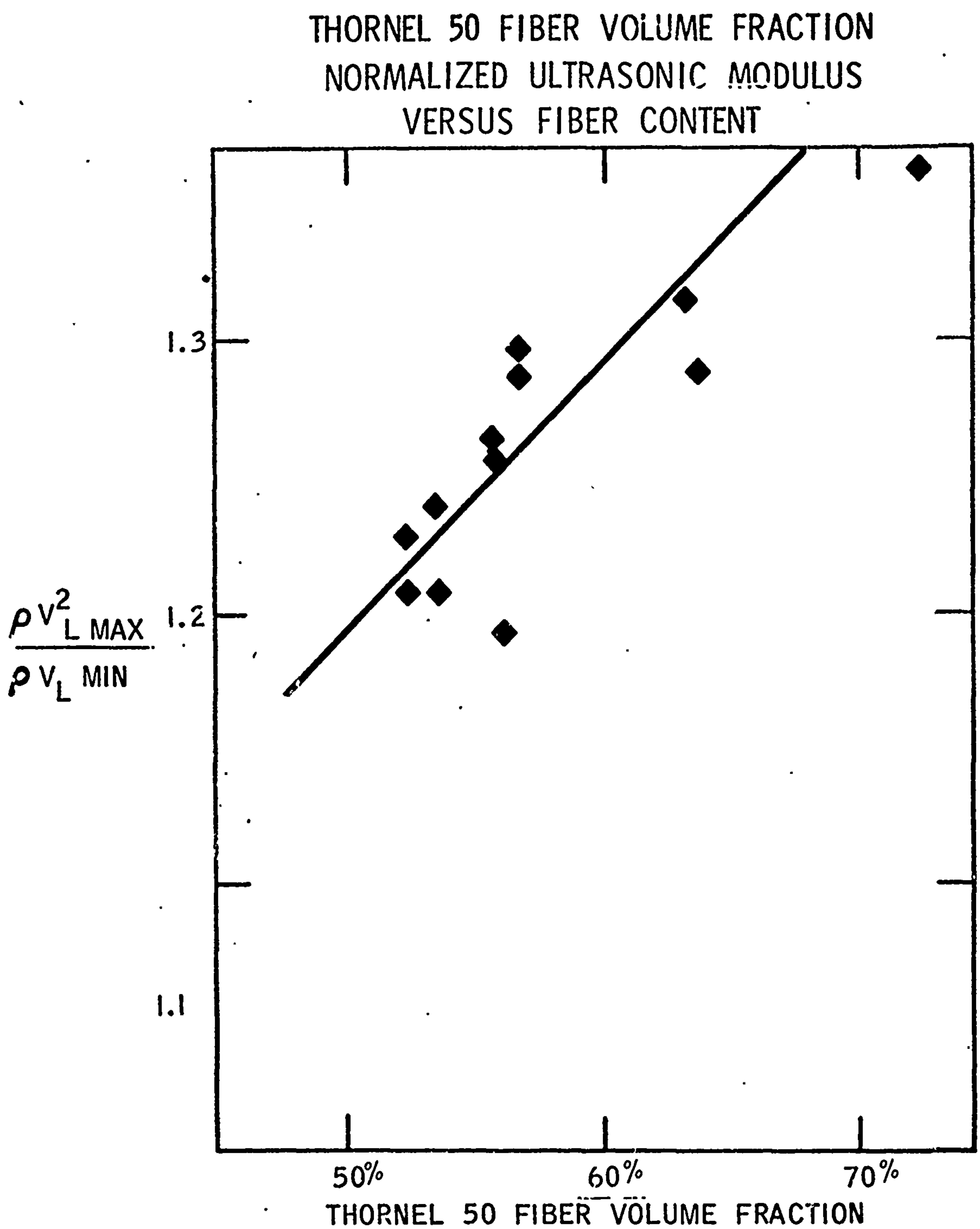

FIGURE 2 EFFECT OF FIBER VOLUME FRACTION ON THE NORMALIZED ULTRASONIC MODULUS

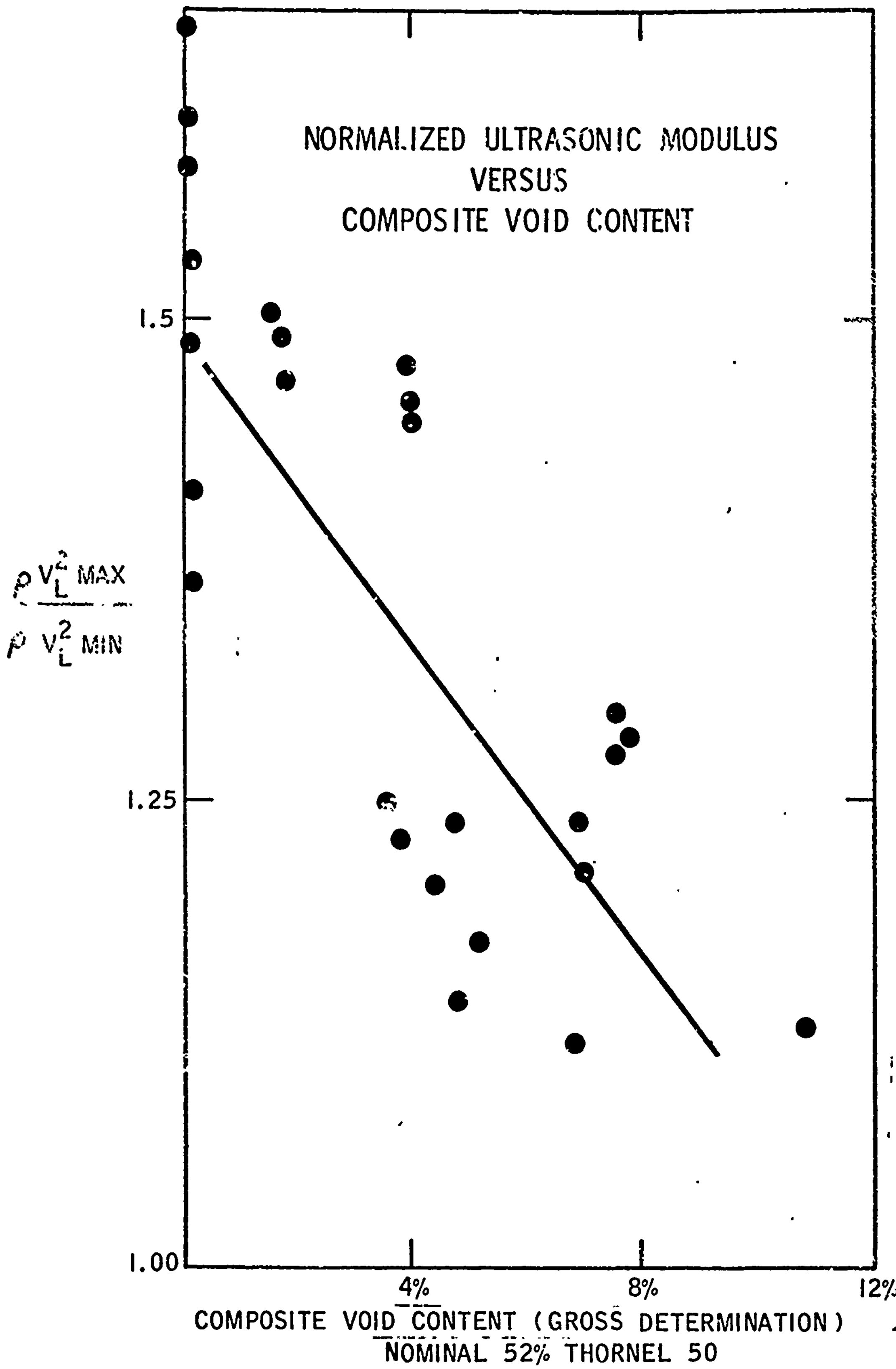

FIGURE 3 EFFECT OF THE COMPOSITE VOID CONTENT ON THE NORMALIZED ULTRASONIC MODULUS (THORNEL 50/EPOXY - 52% V_F)

In summary, our approach to voids control has been to survey the possible ranges of porosity which might be encountered in practice; to thoroughly characterize these voids; to systematically establish the relative effects of such voids on mechanical behavior; to estimate the influence of voids on structural laminates in order to determine allowable void content; and finally, to establish empirical nondestructive, destructive, and void content interrelationships which would allow monitoring the degree of porosity in the laminate under consideration.

SECTION III

3.0 EFFECT OF VOIDS ON MECHANICAL PROPERTIES

3.1 Quality Control Effects

3.1.1 Effect of Voids on the Resin

Resin plates were prepared using the same prepreg resin as for the Thornel 50/epoxy composites. Mechanical stirring and a two-day resin advancement were used to induce void contents ranging from 0 to 9%.

Void content was determined by a mercury displacement specific gravity technique. This is a gross porosity measurement and considerable variation in void content is anticipated.

Figure 4 shows the linear variation in wave propagation velocity as a function of void content; and, as expected, due to the relationship between modulus and wave velocity, the tensile and compressive modulus, as shown in Figure 5, vary linearly with void content. The rapid decrease in tensile strength with increasing void content is shown in Figure 6.

3.1.2 Effect of Voids on Graphite/Epoxy Composites

Unidirectional laminates having a nominal volume fraction of 52% were fabricated using Thornel 50/epoxy and Modmor II/5206. A range of void contents estimated to vary from 0 to 11% was introduced by press molding to various stops. The panels used are described in Table 1.

Accurate determinations of the void content in the Thornel 50/epoxy specimens were made using point-count metallography techniques. More than 100 photomicrographs were obtained on specimens (Thornel 50/epoxy) previously discussed in Reference (1). These photographs were subjected to the statistical void content characterization techniques presented in Reference (2). Detailed information on the number, size and location of voids was obtained by means of a fine grid superimposed on the photomicrographs, and the resulting information served as input data for an elementary computer program. The computer calculated the average and local void contents, based on the imposed grids. Nearest void interaction distances were also tabulated. Thus, by applying the computer technique, the void content variations could be thoroughly characterized. A typical photomicrograph of a high porosity specimen appears in Figure 7. Such information was used to reassess the destructive data and nondestructive measurements previously obtained for the Thornel 50/epoxy composite.

Figures 8 to 11 describe the shear strength, the transverse flexural modulus, the longitudinal flexural strength, and the transverse flexural strength respectively, as a function of void content.

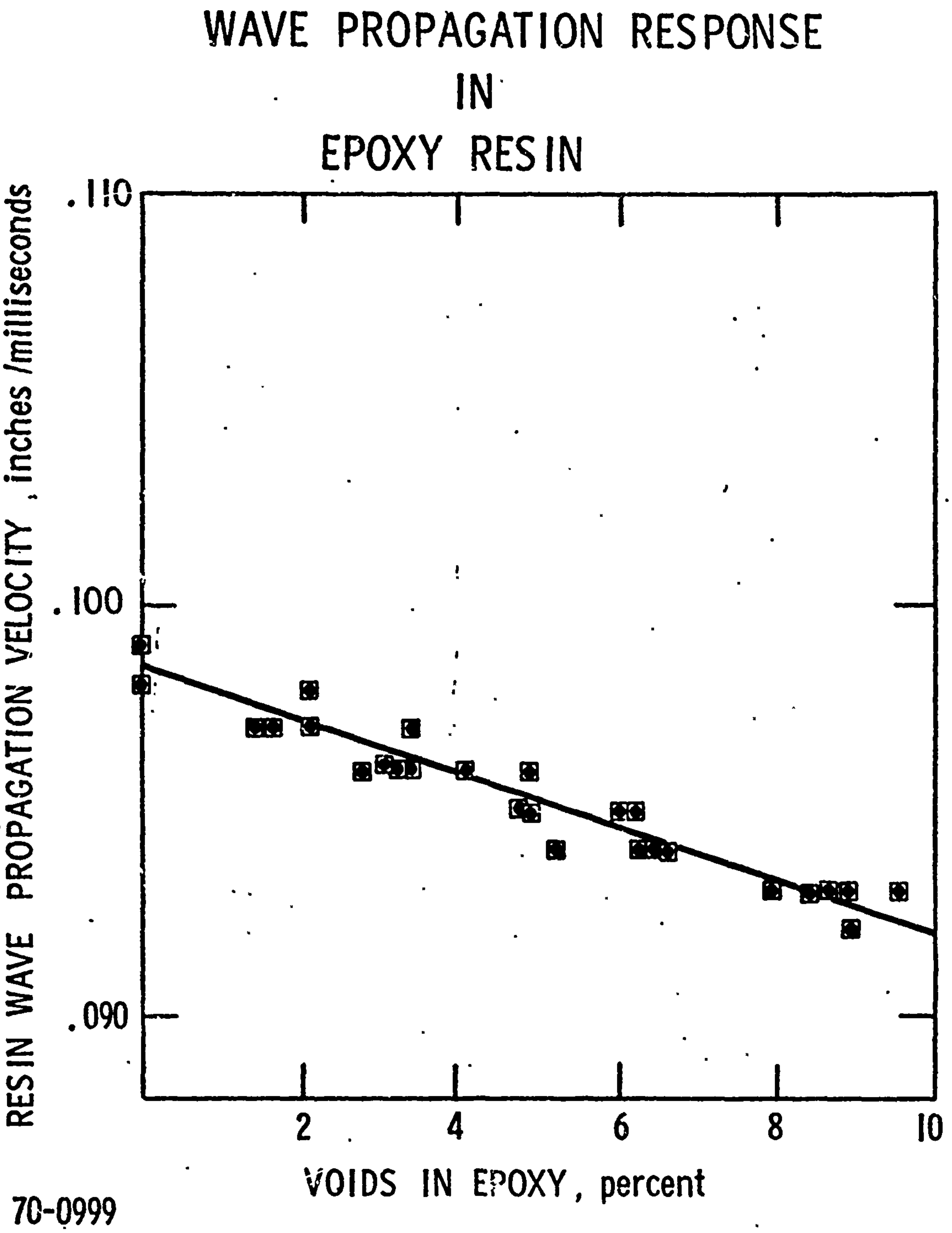

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FIGURE 4 EFFECT OF VOID CONTENT ON THE WAVE PROPAGATION VELOCITY IN EPOXY (THE SAME RESIN AS IN THE THORNEL 50/EPOXY SYSTEM)

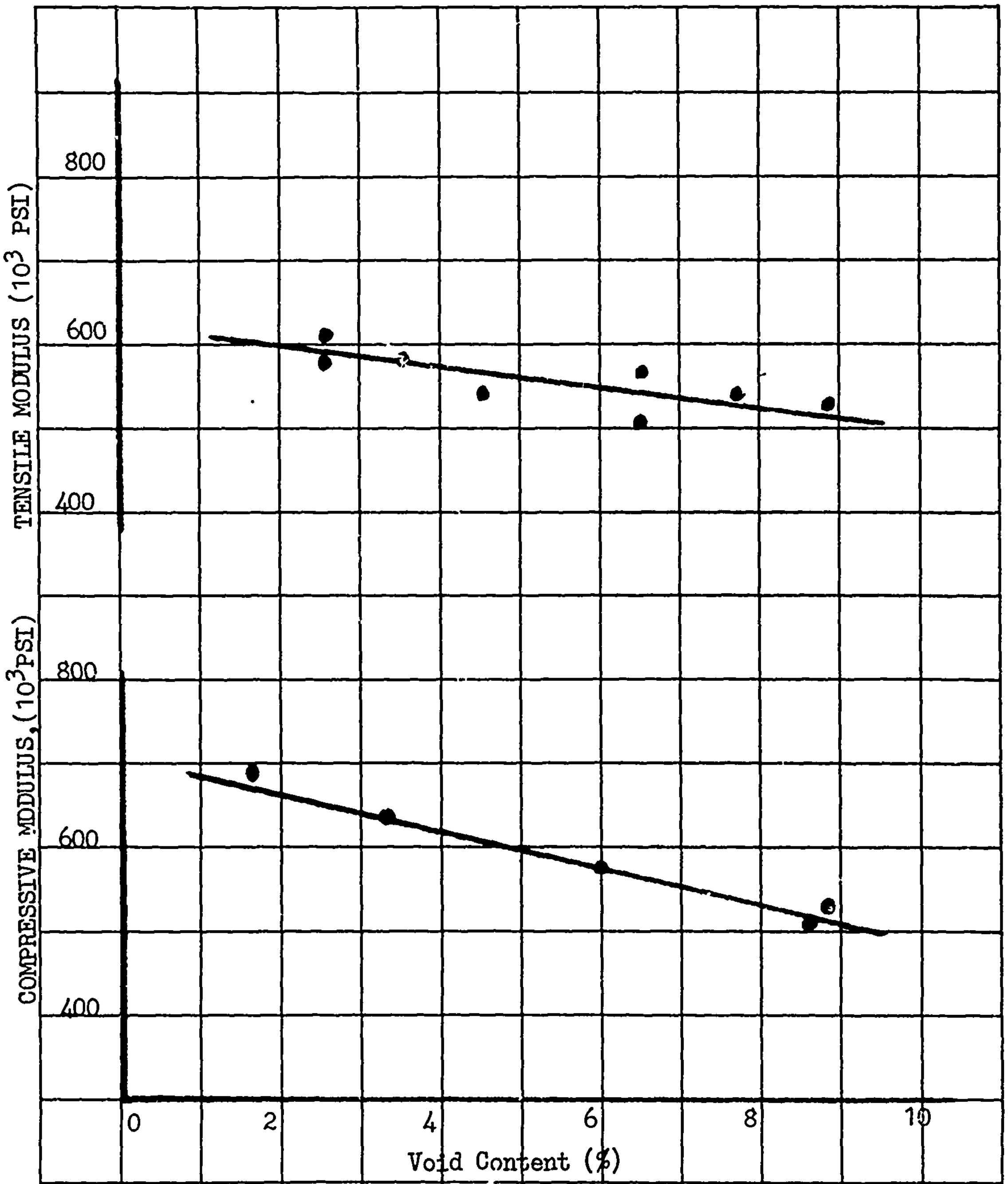

FIGURE 5 EFFECT OF VOID CONTENT ON TENSILE AND COMPRESSIVE MODULUS OF EPOXY (SAME RESIN AS IN THE THORNEL 50?EPOXY COMPOSITE)

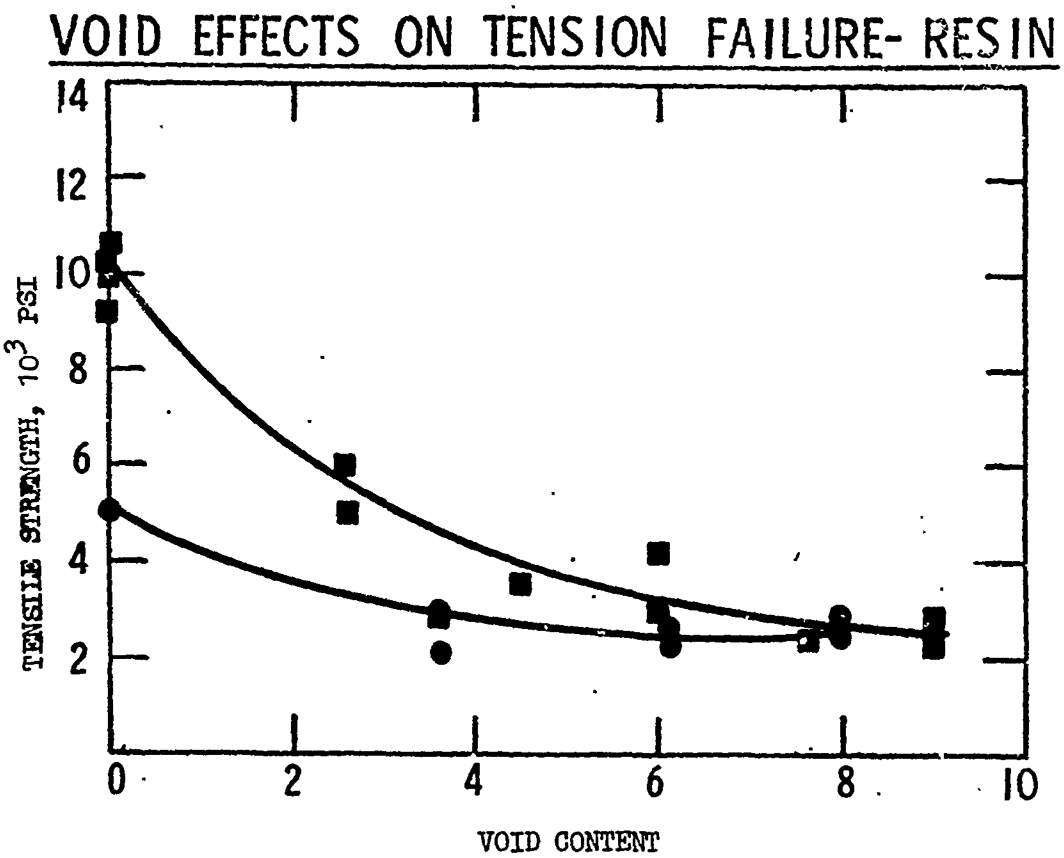

FIGURE 6 EFFECT OF VOID CONTENT ON THE TENSILE STRENGTH OF EPOXY (SAME RESIN AS IN THE THORNEL 50/EPOXY COMPOSITE)

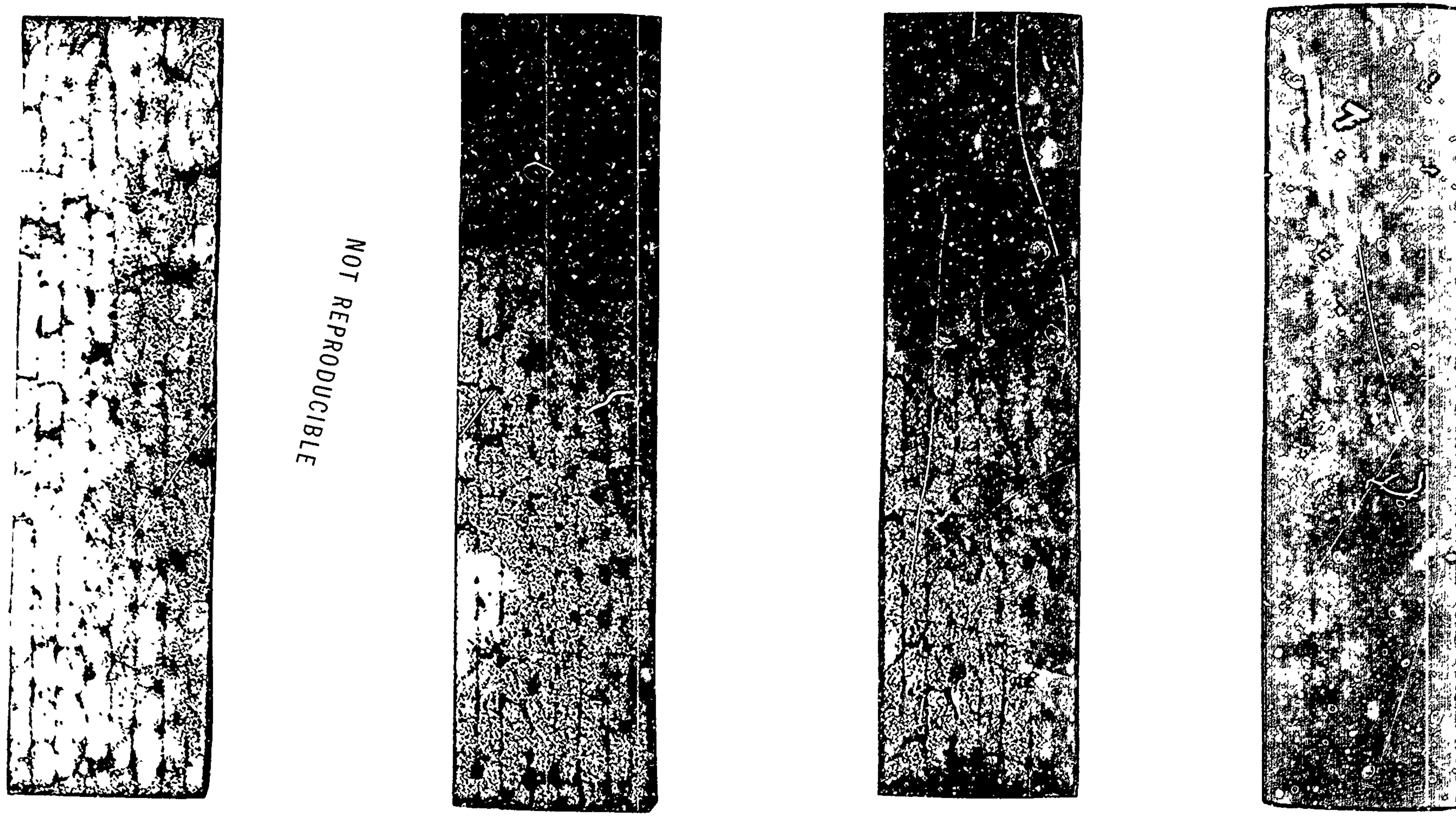

FIGURE 7 TYPICAL PHOTOMICROGRAPH OF A HIGH VOID CONTENT THORNEL 50/ EPOXY SPECIMEN (SECTION TAKEN NORMAL TO THE FIBER AXIS)

Figures 8 to 11 illustrate the fact that the sensitivity of the NDT-voids and mechanical properties interrelationships is greatly enhanced by the use of accurate void characterization.

Figure 12 describes the degradation of these mechanical properties with increases in void content.

To date, void contents for the Modmor II/5206 specimens have been determined using gravimetric techniques and until a point count analysis is performed yielding a more accurate estimate of the amount of voids, the results must be viewed as trends to be verified. Figure 13 shows the percent degradation in various mechanical properties as a function of estimated void content.

Some preliminary NDT measurements have been made, and Figure 14 shows the variation in longitudinal and transverse velocities as a function of void content.

3.2 Effects on Preliminary Design Allowables

3.2.1 Unidirectional Response

In addition to the 4.0 x 4.0-inch panels which were used in the quality control tests, 9.0 x 9.0-inch panels and 3/16-inch diameter rods of unidirectionally reinforced Thornel 50 and Modmor II/epoxy composites were also prepared at high- and low-porosity contents. These panels were machined into longitudinal and transverse tension coupon specimens, and the rods were used to prepare torsion specimens. The experimental results obtained are described on the following pages.

Longitudinal Tension

Longitudinal tension tests were performed using straight sided 9 inch long by 1/2 inch wide specimens. Glass epoxy tabs were bonded to the specimen ends to minimize gripping stresses. Testing was carried out at room temperature and at 250°F on high and low porosity specimens of Thornel 50/epoxy or Modmor II/5206. The results are summarized in Table 2.

Transverse Tension

Transverse tension specimens were 4.5 inches long and had bonded fiberglass epoxy load spreading tabs. The data are presented in Table 2.

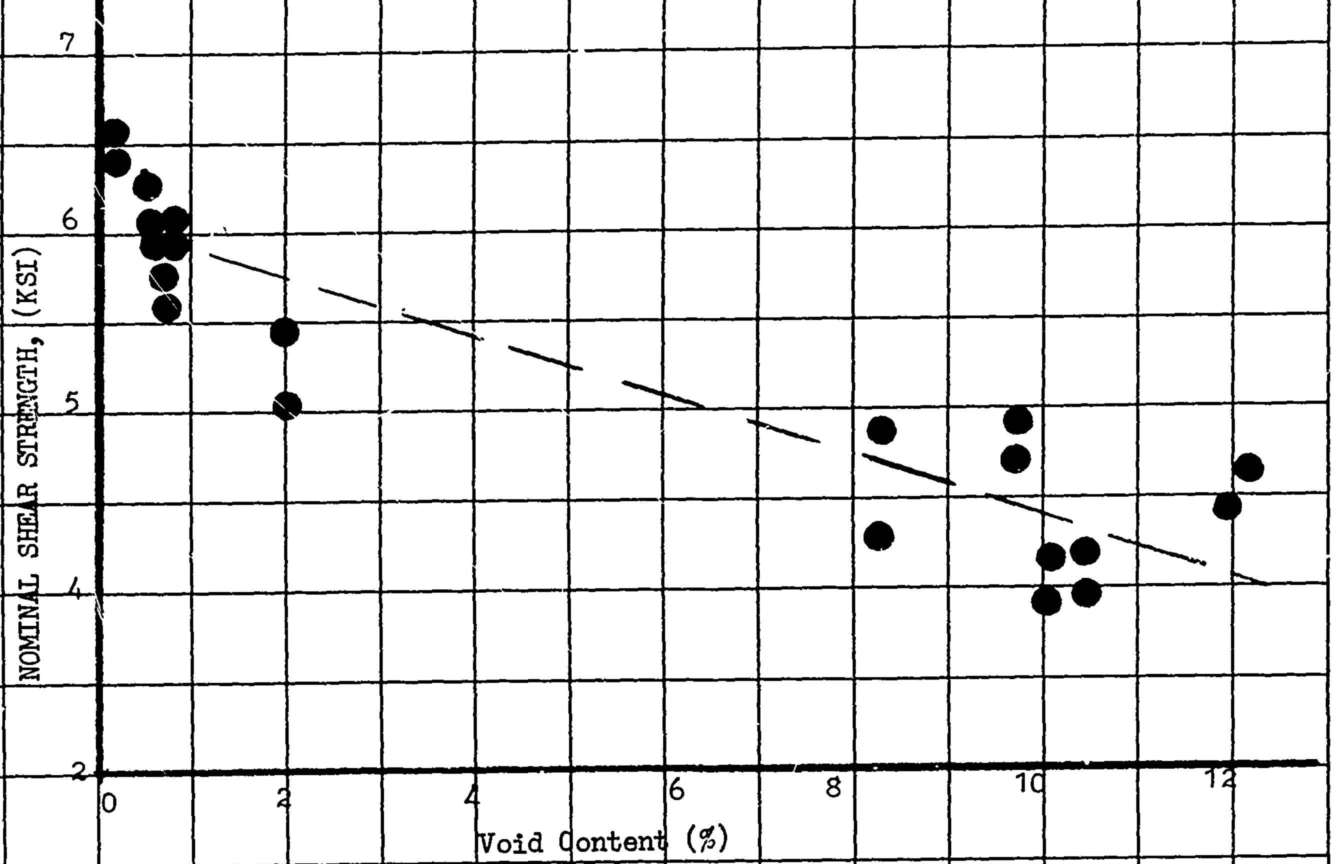

FIGURE 8 EFFECT OF VOID CONTENT ON THE SHEAR STRENGTH OF THORNEL 50/EPOXY (52% V_F)

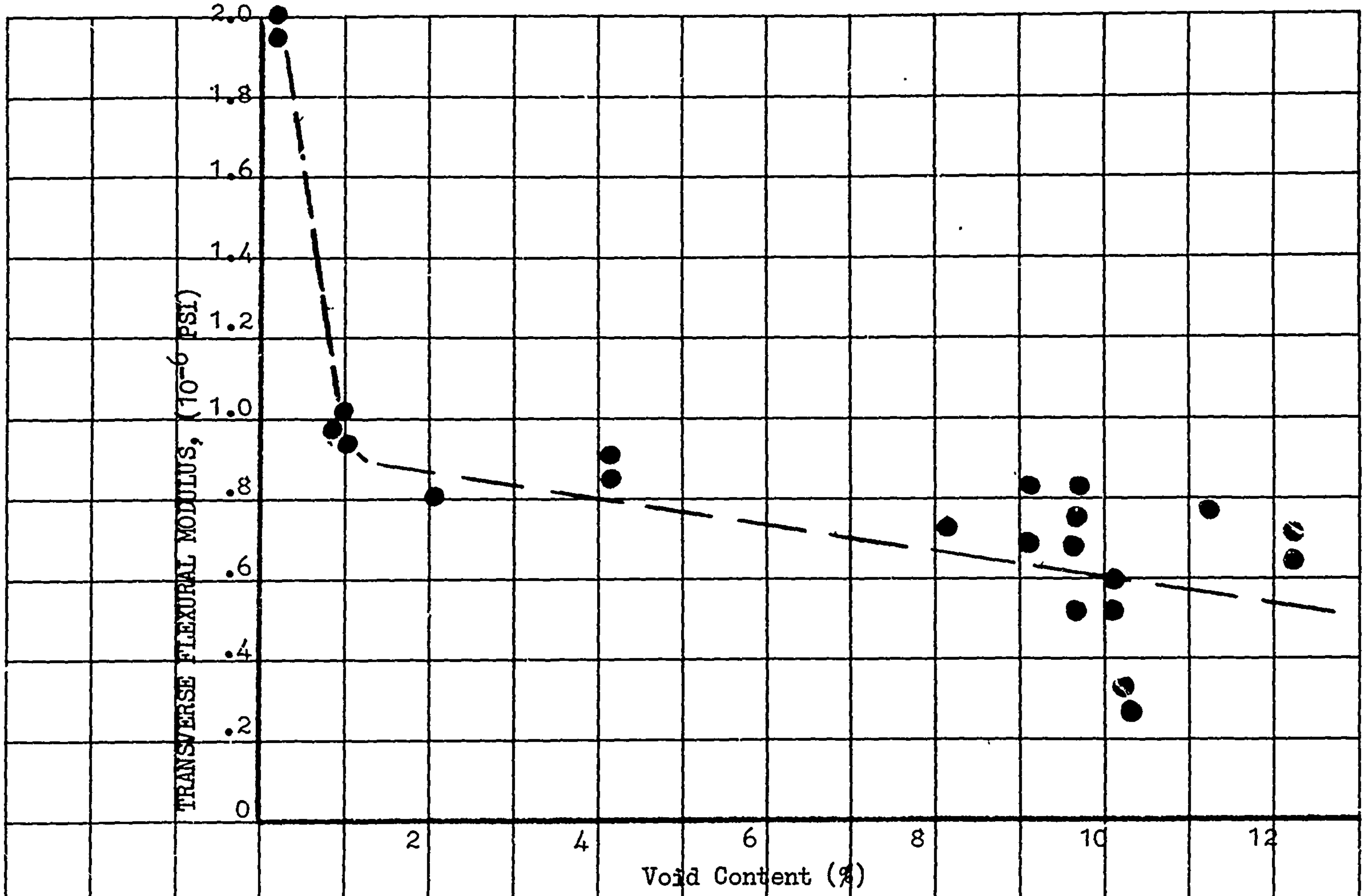

FIGURE 9 EFFECT OF VOID CONTENT ON THE TRANSVERSE FLEXURAL MODULUS OF THORNEL 50/ EPOXY (52% V_F)

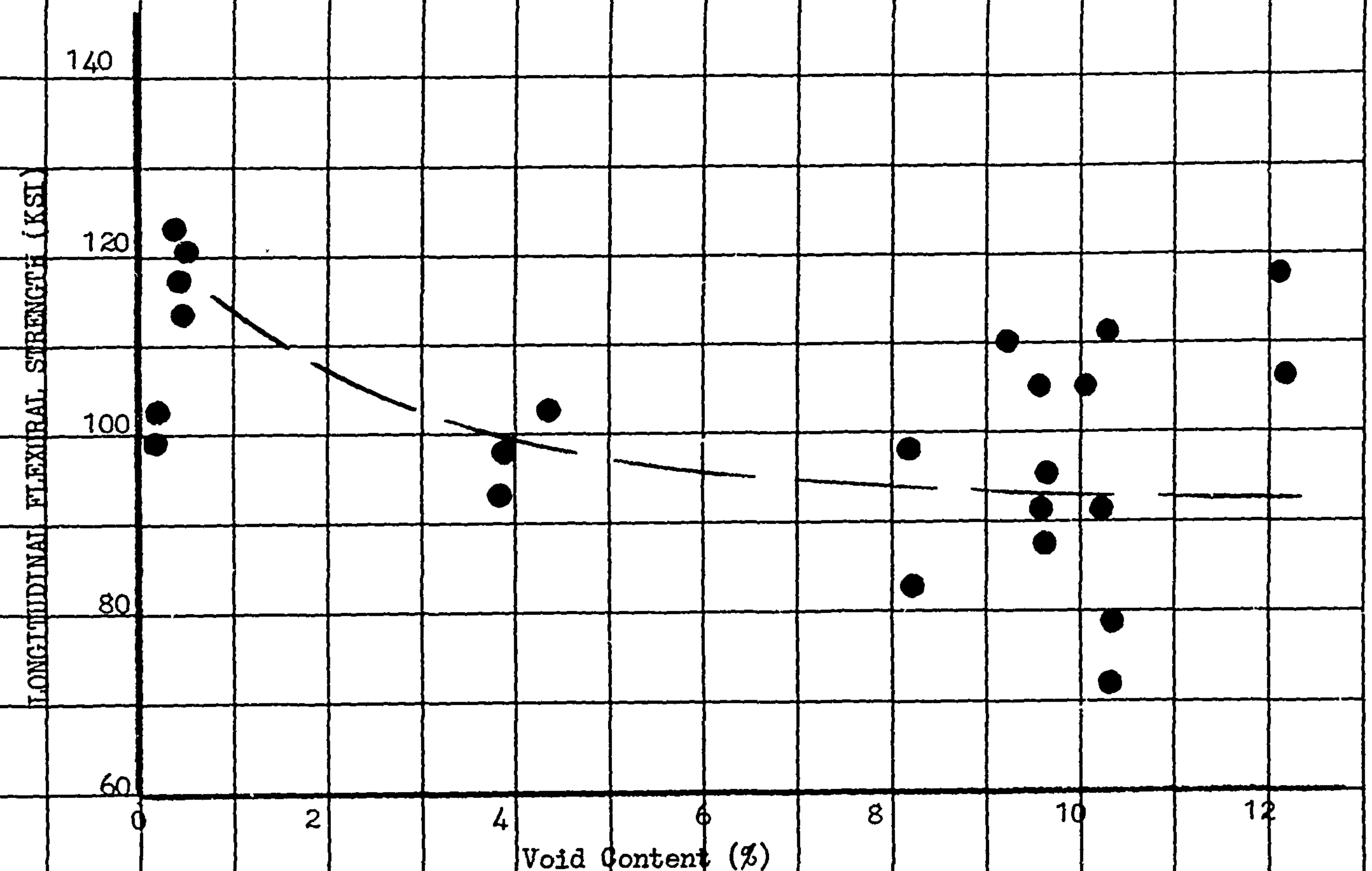

FIGURE 10 EFFECT OF VOIDS ON THE LONGITUDINAL FLEXURAL STRENGTH OF THORNEL 50/EPOXY (52% V_F)

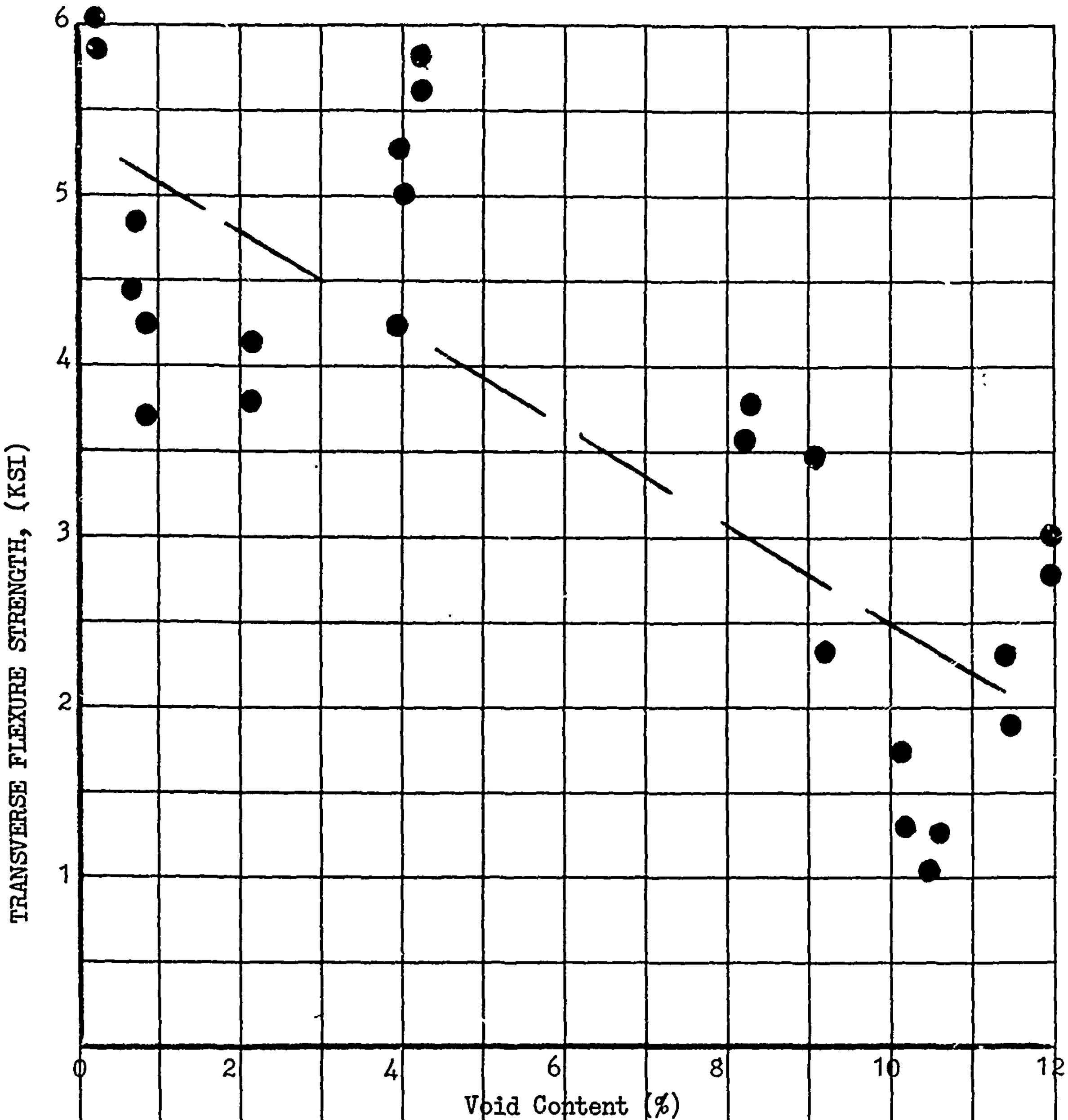

FIGURE 11 EFFECT OF VOIDS ON THE TRANSVERSE FLEXURE STRENGTH OF THORNEL 50/EPOXY (52% V_F)

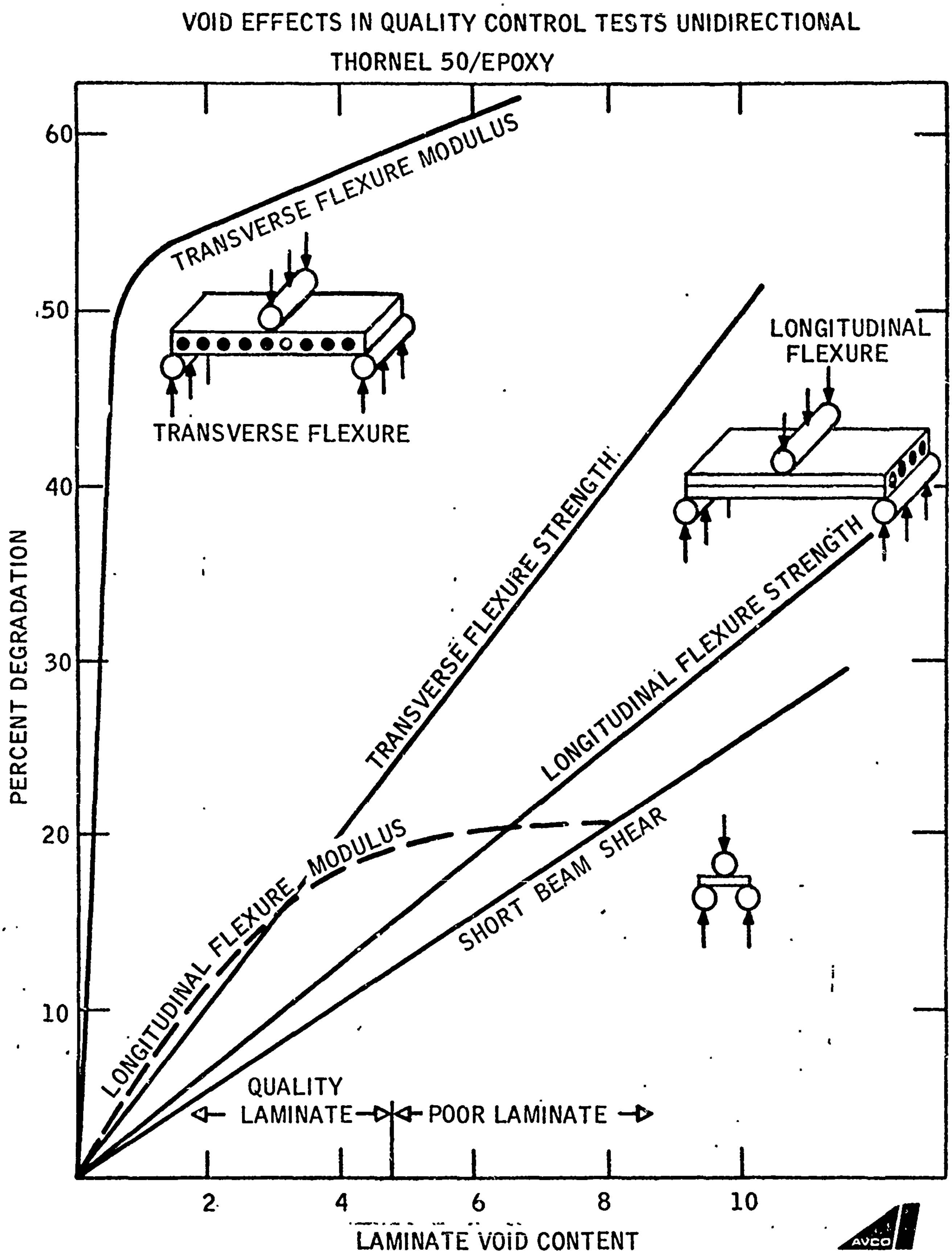

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FIGURE 3 TYPICAL VOIDS EFFECTS IN QUALITY CONTROL TESTS, THORNEL 50/EPOXY (52% VF)

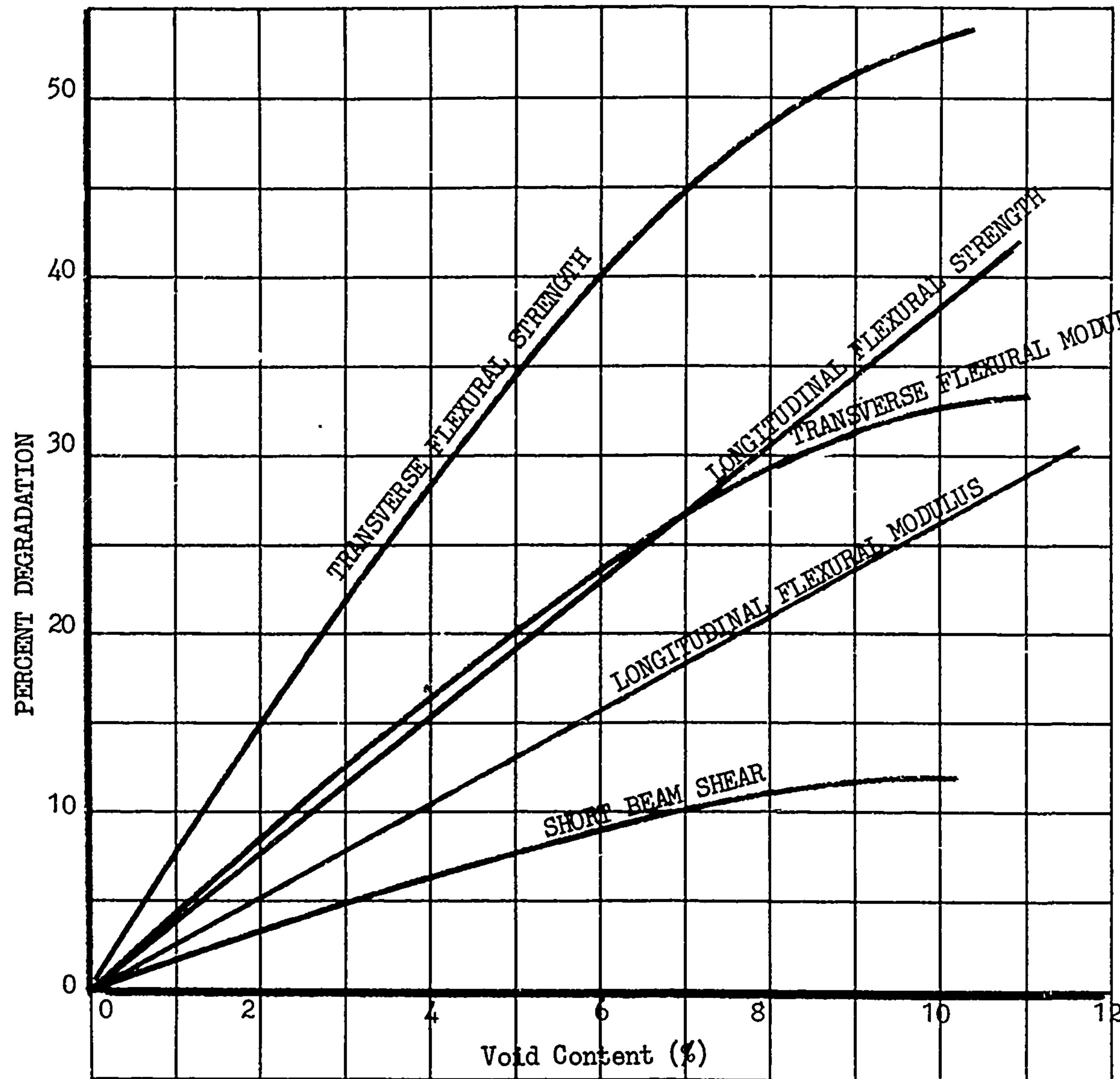

FIGURE 13 TYPICAL EFFECT OF VOIDS IN QUALITY CONTROL TESTS MODMOR II/5206 (52% V_F)

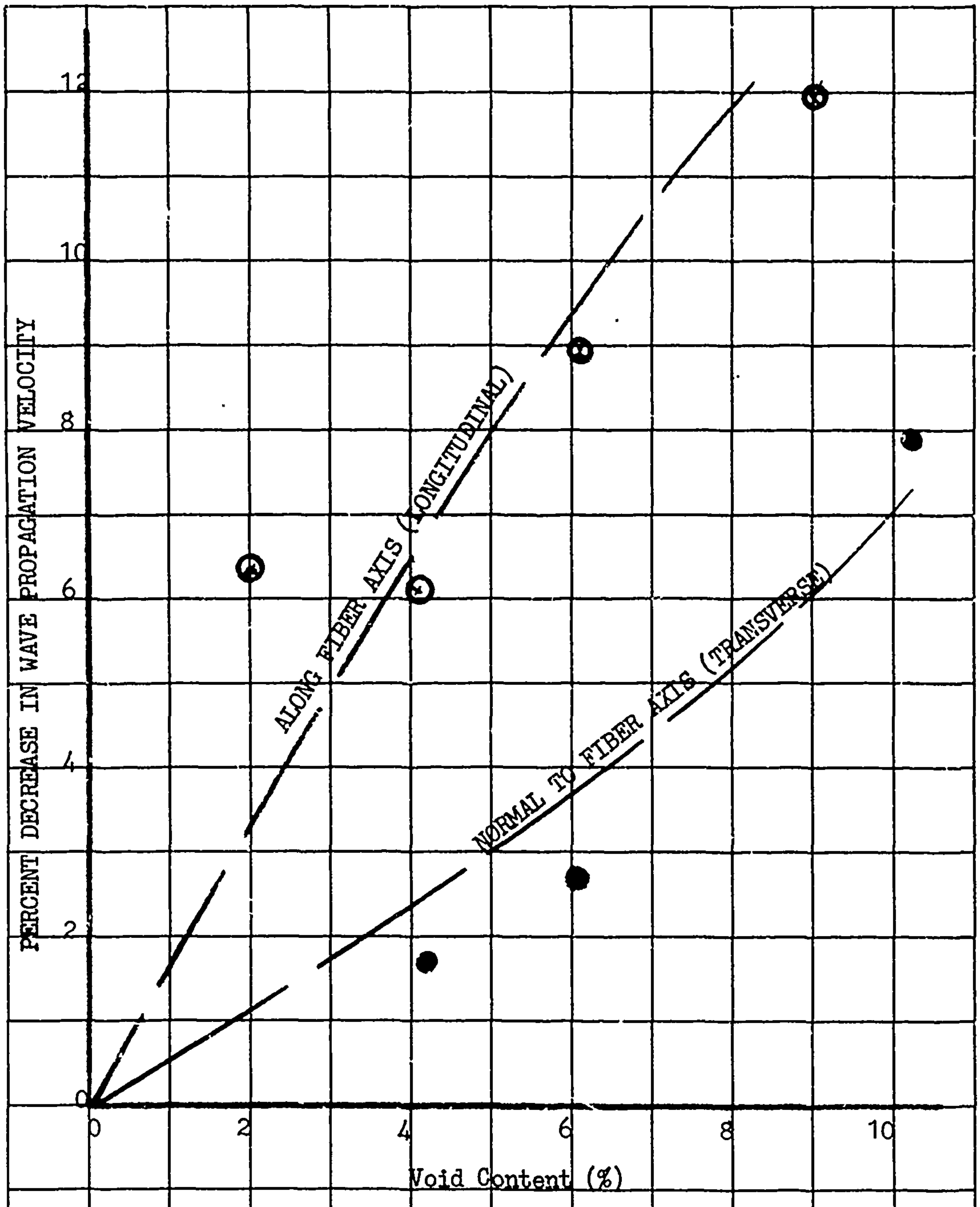

FIGURE 14 EFFECT OF VOID CONTENT ON THE WAVE VELOCITY IN MODMOR II/5206; 52% V_F

TABLE 1 - SUMMARY OF GRAPHITE EPOXY PLATES AND RODS

Panel No.	Layup	Panel Size, in.	Panel Thickness, in.	Panel Density, g/cc	Estimated Wgt. Resin Content	Porosity
Morganite 11/Narmco 5206 Epoxy*(Modulite 5206)						
1117-25	0°	4 x 4	.077	1.346	29.5	---
-26	0°	4 x 4	.058	1.512	22.2	---
-29.	0°	9 x 9	.055	1.551	20.0	Low
-30	0°	4 x 4	.059	1.487	25.6	High
-31	0°	4 x 4	.064	1.450	27.0	High
-32	0°	4 x 4	.068	1.408	30.5	----
-37	$0^{\circ}/90^{\circ}/0^{\circ}/\pm45^{\circ}$	9 x 9	.065	1.44	28.0	High
-40	0°	9 x 9	.065	1.430	27.8	----
-43	$0^{\circ}/90^{\circ}/0^{\circ}/+45^{\circ}$	9 x 9	.056	1.520	23.8	Low
-45	$0^{\circ}/\pm45^{\circ}/\pm45^{\circ}/90^{\circ}$	9 x 9	.058	1.471	24.1	High
Rods (3/16" Diameter)						
1117-39	0°	9	.187	1.34		
-42	0°	9	.194	1.62		
-47	0°	9	.174	1.52		
Thornel 50/Polaris Epoxy **						
1117-28	0°	9 x 9	.055	1.504	29.3	Low
-36	0°	9 x 9	.065	1.427	38.3	High
-38	$0^{\circ}/90^{\circ}/0^{\circ}/+45^{\circ}$	9 x 9	.066	1.41	39.7	High
-41	$0^{\circ}/90^{\circ}/0^{\circ}/\mp45^{\circ}$	9 x 9	.066	1.48	37.8	Low
-46	0°	4 x 4	.060	1.401	25.8	High
-49	$0^{\circ}/+45/-45/90^{\circ}$	4 x 4	.060	1.410	29.7	High
Rods (3/16" Diameter)						
1117-44	0°	9	.179	1.388		
	0°	9	.179	1.469		
	TOTALS	21 Items				

* Panel Resin Content Estimate Based on 40% nominal resin content.
** Panel Resin Content Estimate Based on 42% nominal resin content.

Torsion

Experiments were completed on 3/16 inch diameter rods of 3.5 inch length. The rods were bonded into 1.0 inch long aluminum load blocks which had previously been drilled .005 inches over size to accommodate the composite specimens. Foil-loaded strain gages were employed to measure the torque versus twist response of the rods. Experiments were conducted at room temperature. Typical torque versus shear strain response is shown in Figures 15 and 16, and the resulting torsion strengths and shear modulus values are listed in Table 2.

3.2.2 Quasi-Isotropic Behavior

Experimentally Determined Properties

Several angle ply orientations and stacking sequences were investigated using both Thornel 50 and Modmor II epoxy composites. These included Panels 1117-37, 43, 45, 38, 41 and 49. Referring to Table 1, these plates represented symmetric and nonsymmetric stacking sequences and high and low porosity composites.

Tension specimens 9.0 inches by 1/2 inch were prepared and tested at 73° and 250°F. The resulting data is presented in Table 3 .

Analytically Determined Properties

The mechanical properties of a Modmor II angle ply laminate were estimated using a laminate analysis (3). The laminate selected for this analytical evaluation was a 10-ply laminate with a quasi-isotropic (0/90/±45°) symmetrical construction. Included in this evaluation was the prediction of the modulus and strength envelopes under off-axis tensile loading at room temperature and 250°F. Two laminates were considered; one with a "low porosity", the second with a "high porosity".

In general, the laminate program calculates the in-plane extensional and shear stiffnesses, as well as the average stress in the laminate assuming various criteria for failure. The calculated in-plane extensional (A_{11}) and shear (A_{66}) stiffness coefficient are defined in terms of the loading axes (α and β) as:

$$A_{11} = \frac{E_{\alpha\alpha}}{1-\mu_{\alpha\beta}\mu_{\beta\alpha}}$$

and

$$A_{66} = G_{\alpha\beta}$$

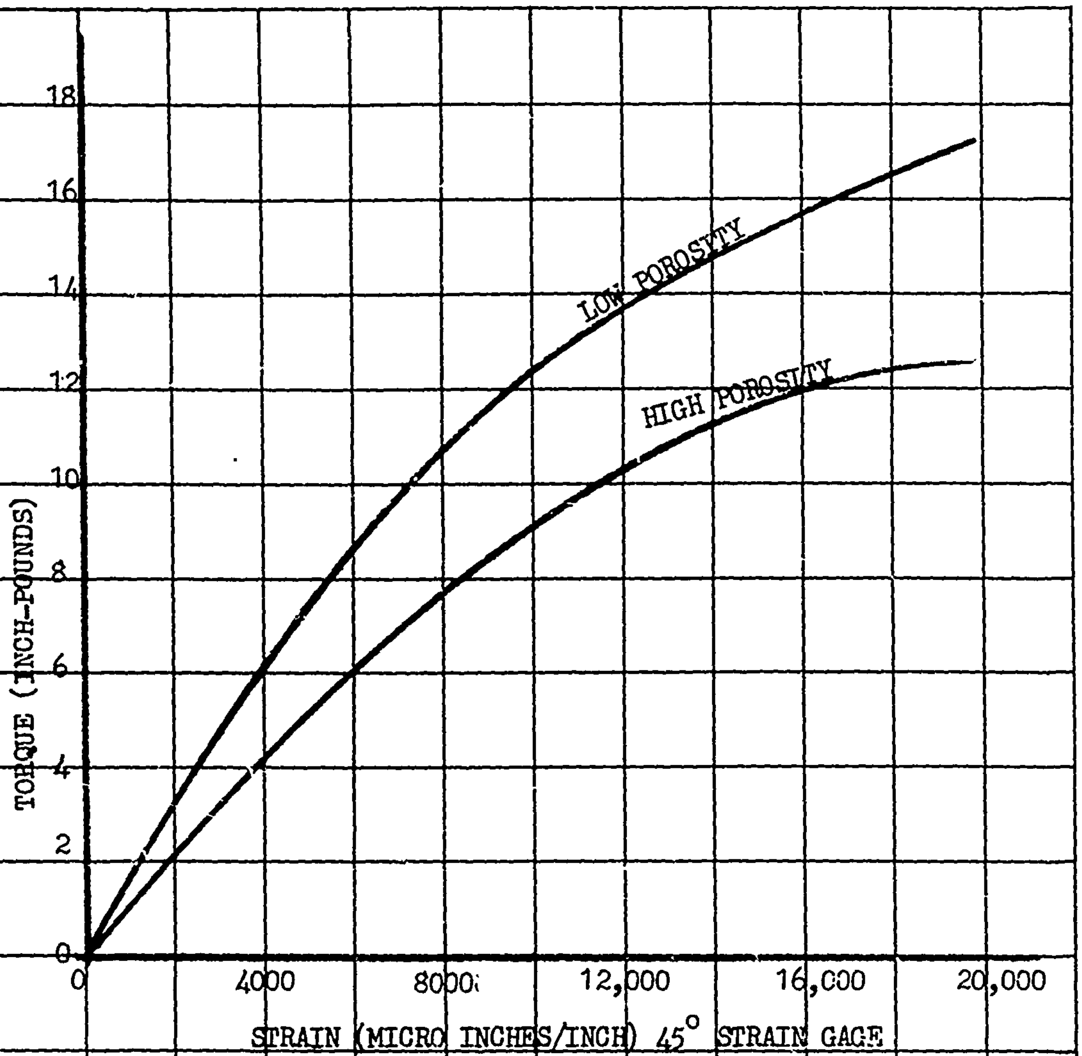

FIGURE 15 TYPICAL TORSIONAL RESPONSE OF MODMOR II/5206, 52% V_F

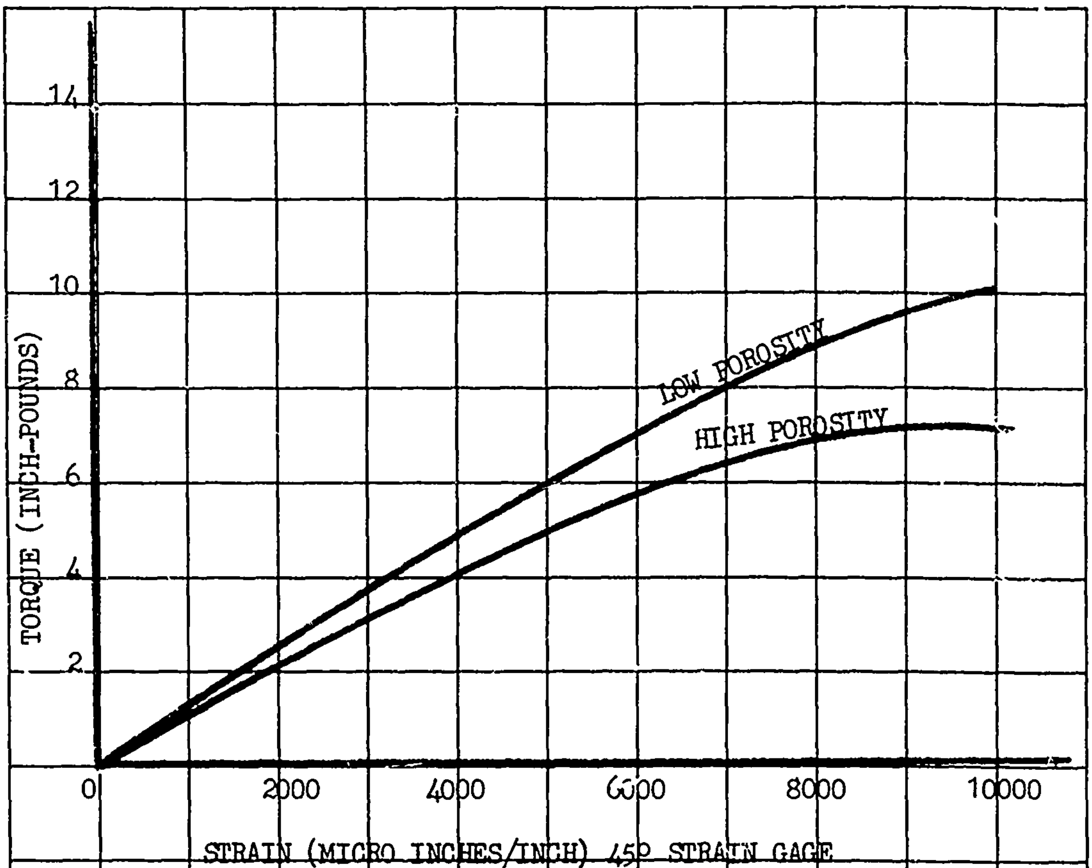

FIGURE 16 TYPICAL TORSIONAL RESPONSE OF THORNEL 50/EPOXY, 52% V_F

TABLE 2

VOID EFFECTS ON PRELIMINARY DESIGN ALLOWABLES

Property	Room Temperature				250°F			
	Thornel 50/Epoxy		Modmor II/5206		Thornel 50/Epoxy		Modmor II/5206	
	Low P	High P	Low P	High P	Low P	High P	Low P	High P
Long. Modulus $E_{11} \times 10^{-6}$	30.2	26.8	25.7	21.2	29.5	27.4	23.8	22.2
Trans. Modulus $E_{22} \times 10^{-6}$	.80	.85	1.17	1.12	.079	.093	1.37	.92
Shear Modulus $G_{12} \times 10^{-6}$	.584	.476	.79	.546				
Poisson's Ratio ν_{12}	.33	.36	.37	.34	.44	.51	.36	.33
Poisson's Ratio ν_{21}	.016	.018	.035	.029	.016	.022	.028	.026
Trans. Total Strain	.24	.30	.40	.37	.55	.63	.38	.42
Long. Total Strain	.40	.38	.89	.61	.27	.23	.79	.55
Long. Strength	121000	105000	239000	128000	75000	60400	191000	115000
Trans. Strength	1910	2520	5740	3980	513	598	4650	3790
Torsion Strength	8200	5900	13000	10000				

TABLE 3

QUASI-ISOTROPIC LAMINATE RESPONSE

Property	Room Temperature					250°F				
	Thornel 50/Epoxy		Modmor II/5206			Thornel 50/Epoxy		Modmor II/5206		
	LP	HP	LP	HP	HP --ODD	LP	HP	LP	HP	HP - ODD
Plate	1117-41	1117-38	1117-03	1117-37	1117-45	1117-41	1117-38	1117-03	1117-37	1117-45
Ultimate Stress	52.6	71.7	100.0	72.9×10^3	56.8	32.3	27.1	96.2	76.3	54.2
Ultimate Strain	.42	.48	.78	.66	.70	.32	.19	.79	.70	.63
Modulus	13.7	14.8	12.5	11.1×10^6	8.45	12.0	13.7	12.1	10.8	8.37
Poisson's Ratio	.18	.16	.20	.20	.33	.10	.14	.19	.23	.34

The shear modulus (G) as a function of angle is therefore a direct output of the program; however, the extensional modulus E requires knowledge of the two Poisson's ratios ($\mu_{\alpha\beta}$ and $\mu_{\beta\alpha}$), or more specifically, their product. For an angle ply laminate of the construction considered here, this product can be assumed to be a constant (i.e. independent of loading angle).

Using the experimental values obtained on unidirectionally reinforced epoxy (Modmor II), analytical predictions were made of the strength and stiffness envelopes at 75° and 250°F for a low and high porosity material. The properties used for these predictions are given in Table 5. (Note: The shear modulus and strength values at 250°F are estimated and were scaled according to the increased temperature sensitivity of the transverse (90°) tensile data.)

Figure 17 is a plot of the predicted tension and shear moduli as a function of loading angle for the two temperature and porosity levels considered. Curves (1) and (2) are the predicted moduli at 75° and 250°F for low porosity material. These two curves have the general characteristics one would expect, and their positions with respect to each other are as would be expected (i.e. the elevated temperature curve lies below the room temperature curve.).

Curves (3) and (4) are for the high porosity material. They exhibit the same general characteristics and are in the same relative position (with respect to the low porosity curves) one would expect; however, the elevated temperature modulus lies above the room temperature curve. This is due to the input data (see Table 4) where the elevated temperature modulus is about 5% higher than the room temperature value. This may be characteristic of the high porosity material; however, at this point, the exact void level in each sample is not known and this reversal may be a manifestation of differences in actual void content.

As mentioned earlier, the laminate analysis program calculates the average stress in the laminate assuming various modes of failure. The program calculates the average stress required to fail the laminates at each orientation. No allowance is made for successive laminate failure. For the constructions considered here, four strength envelopes are calculated; one for each of the orientations (0°, 90°, +45°, -45°). Figure 18 is a plot of these various strength envelopes for the low porosity materials at 75°F. One then must define a mode or criteria for failure. One could define failure as the failure of any laminate. The failure envelope, then, would be established by the lower bound of the curves of Figure 18. When loaded at 0°, failure (as defined above) will occur in the 90° laminates. When loaded at +45°, the failure is in the -45° laminates; and when loaded at 90°, the failure will be in the 0° laminates, and the "failure stress" would vary from 60 Ksi at 0° to 40 Ksi at 90°.

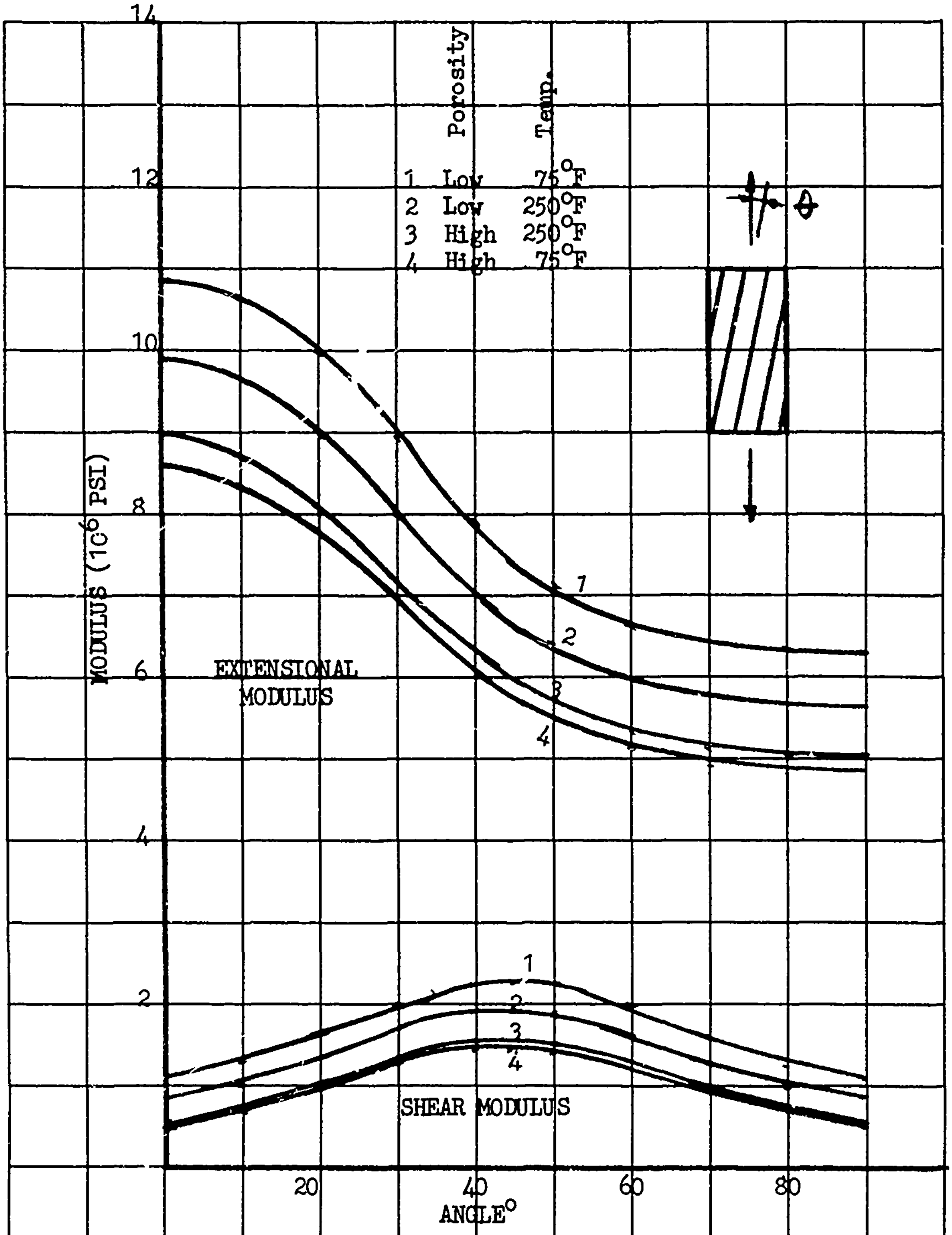

FIGURE 17 PREDICTED VARIATION IN THE MODULI OF A UNIDIRECTIONAL GRAPHITE EPOXY COMPOSITE AS A FUNCTION OF FIBER ORIENTATION

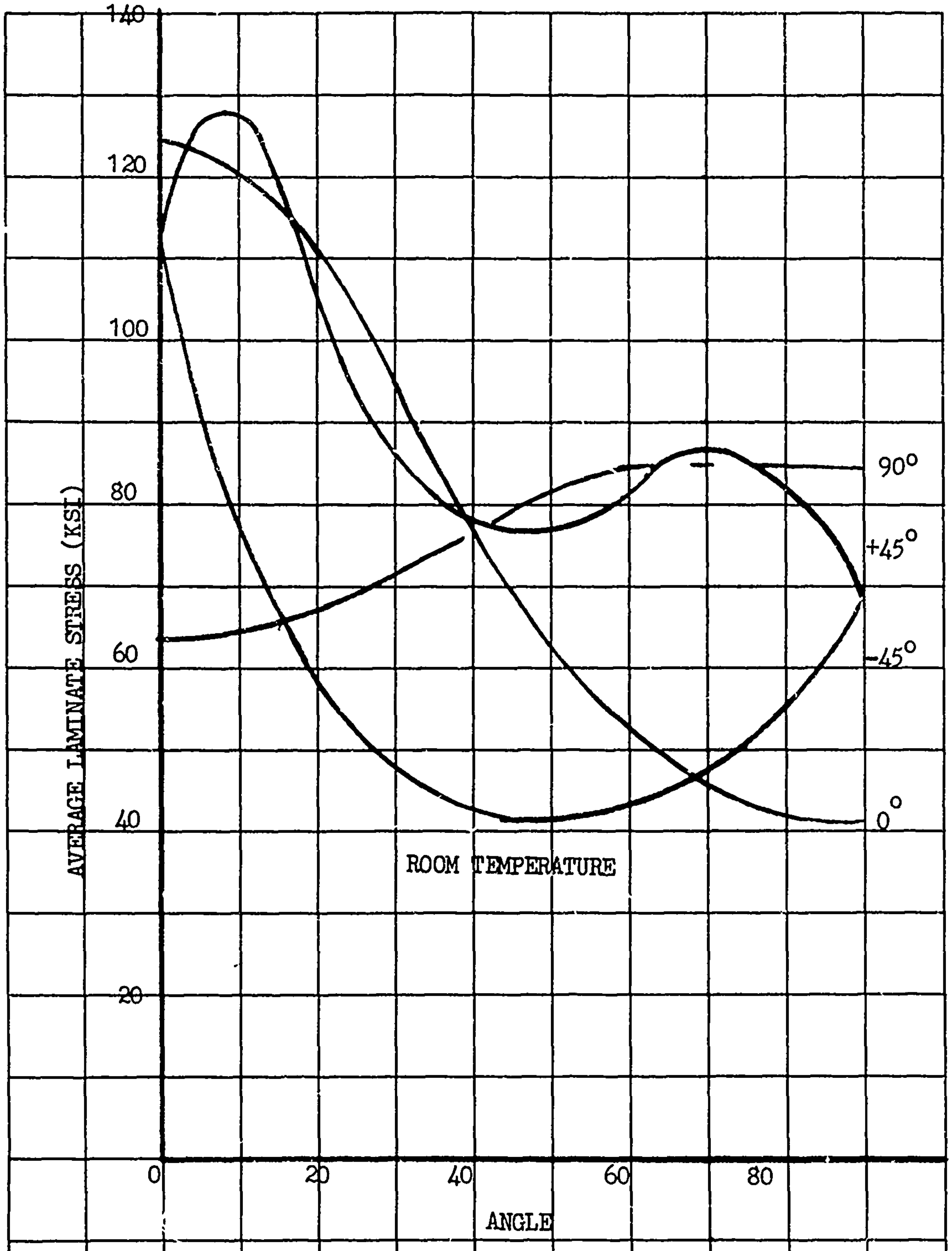

FIGURE 18 PREDICTED FAILURE ENVELOPES FOR INDIVIDUAL PLYS OF A LOW POROSITY GRAPHITE/EPOXY LAMINATE (0/90/0/±45° LAYUP)

TABLE 4

UNIDIRECTIONAL PROPERTIES FOR MODMOR TYPE II

Porosity / Test Temperature / Property	RT		250°F	
	Low	High	Low	High
E_{11} (10^6 psi)	25.7	21.2	23.8	22.2
E_{22} (10^6 psi)	1.17	1.12	1.37	.92
G_{12} (10^6 psi)	.79	0.55	(.70)*	(.56)
ν_{12}	.37	.34	.36	.33
ν_{21}	.035	.029	.028	.026
σ_{11} (10^3 psi)	239.	128.	191.	115.
σ_{22} (10^3 psi)	5.7	3.98	4.65	3.79
τ_{12} (10^3 psi)	13.0	10.	(10.)*	(9.5)*

*Estimated

If one were to use the criteria of catistrophic failure (i.e. failure of all the laminates as occurs in a tensile test), one could not use the upper bound as a failure envelope. The predicted maximum stress of 124 KSI when loaded at 0° is based on the assumption that none of the other laminates have failed and are carrying their share of the load. This is not the case. The 90° laminates are predicted to fail at 64 KSI, and the $\pm 45^{\circ}$ laminates should fail at 112 KSI. This corresponds to 60% of the cross-sectional area of the laminate.

In reality, failure (in the classic sense) will occur at some point between the upper and lower bounds. The laminate analysis program is now being modified to include a means of eliminating those laminates which fail and to transfer the load to the remaining laminates. This will provide a means of obtaining a realistic estimate of the strength of angle ply laminates.

Earlier, the tensile properties of Modmor II epoxy laminates with a construction of [0/90/0/±45] were determined at room temperature when loaded in the 0° direction. Again, two laminates were tested, one with a "low porosity" and one with a "high porosity". At this point, it is not known what the exact void levels were or how these levels compared with the unidirectional laminates used for the predictions. However, the comparisons of the predicted and measured moduli is very good, as seen in Table 5 below.

TABLE 5

COMPARISON OF MODULI

Void Level	Modulus (10^6 psi)	
	Predicted	Measured
Low	12.9	12.5
High	11.9	11.1

SECTION IV

4.0 NONDESTRUCTIVE EVALUATION PROCEDURES

A portion of our effort is devoted to the development of correlation between nondestructive and destructive observations. Accordingly, individual specimens were subjected to ultrasonic compressional and shear wave velocity measurements. The next seven illustrations (Figures 19 through 25) present our attempts to seek correlations between NDT measurements and mechanical properties. In order to determine whether significant trends were observable, the data is reduced and presented in terms of normalized ultrasonic modulus, i.e., the observed measurements divided by the minimum value of ρV^2. This should give an indication of the percentage variation of the NDT measurement and thereby give some assurance whether the ultrasonic technique may be used to assess composite materials variability. Let us consider the illustrations in detail.

Figure 19

The ultrasonic data is presented for the individual specimens machined from the unidirectionally reinforced MOD II/5206 Panel 1117-40. In this illustration, we consider the longitudinal tensile modulus and strength. There appears to be a reasonable correlation of modulus, with the normalized ultrasonic modulus variation spanning approximately 5.0%. The relationship with strength is less convincing, however, although there does appear to be a trend.

Figure 20

Here, we are dealing once again with Panel 1117-40; however, now we are comparing the NDT data to the transverse tensile modulus and strength. In this instance, the situation is reversed, as compared to Figure 19 since the strength appears to give a more convincing correlation. The range in ultrasonic variation is quite small, however, being of the order of 3.0% for the individual coupons tested.

Figure 21

The NDT correlations obtained on the torsion rods appear in this illustration. Note that for both the Thornel 50 and MOD II composites, the correlation is quite strong, and variations in the ultrasonics range over 40%. We must realize that in this case we are dealing with five separately fabricated parts, and therefore, considerably more variation in void content and mechanical properties was encountered. The same comment holds true for Figure 23, where data is presented from two plates. The other illustrations under discussion exhibit smaller within-the-plate variability and correspondingly smaller variations in NDT observables.

Referring again to Figure 21 note,that the Thornel 50 exhibits a larger strength variation and better correlation with the NDT data. In part, this is due to the fact that the extreme range of voids in the Thornel 50 was larger than for the MOD II composite.

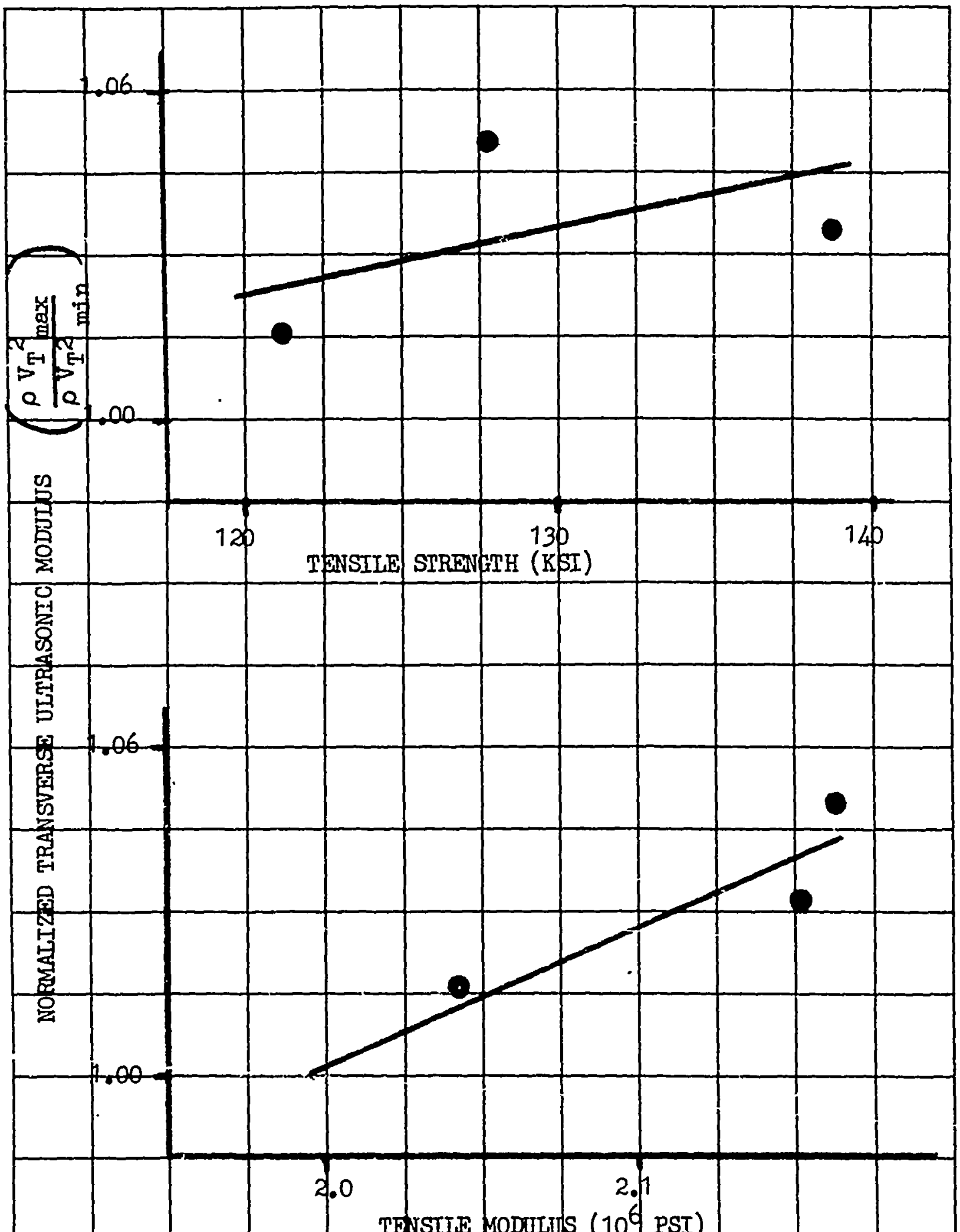

FIGURE 19 NORMALIZED TRANSVERSE ULTRA-SONIC MODULUS VERSUS LONGITUDINAL TENSILE STRENGTH AND LONGITUDINAL TENSILE MODULUS (PLATE 1117-40 MODMOR II/5206)

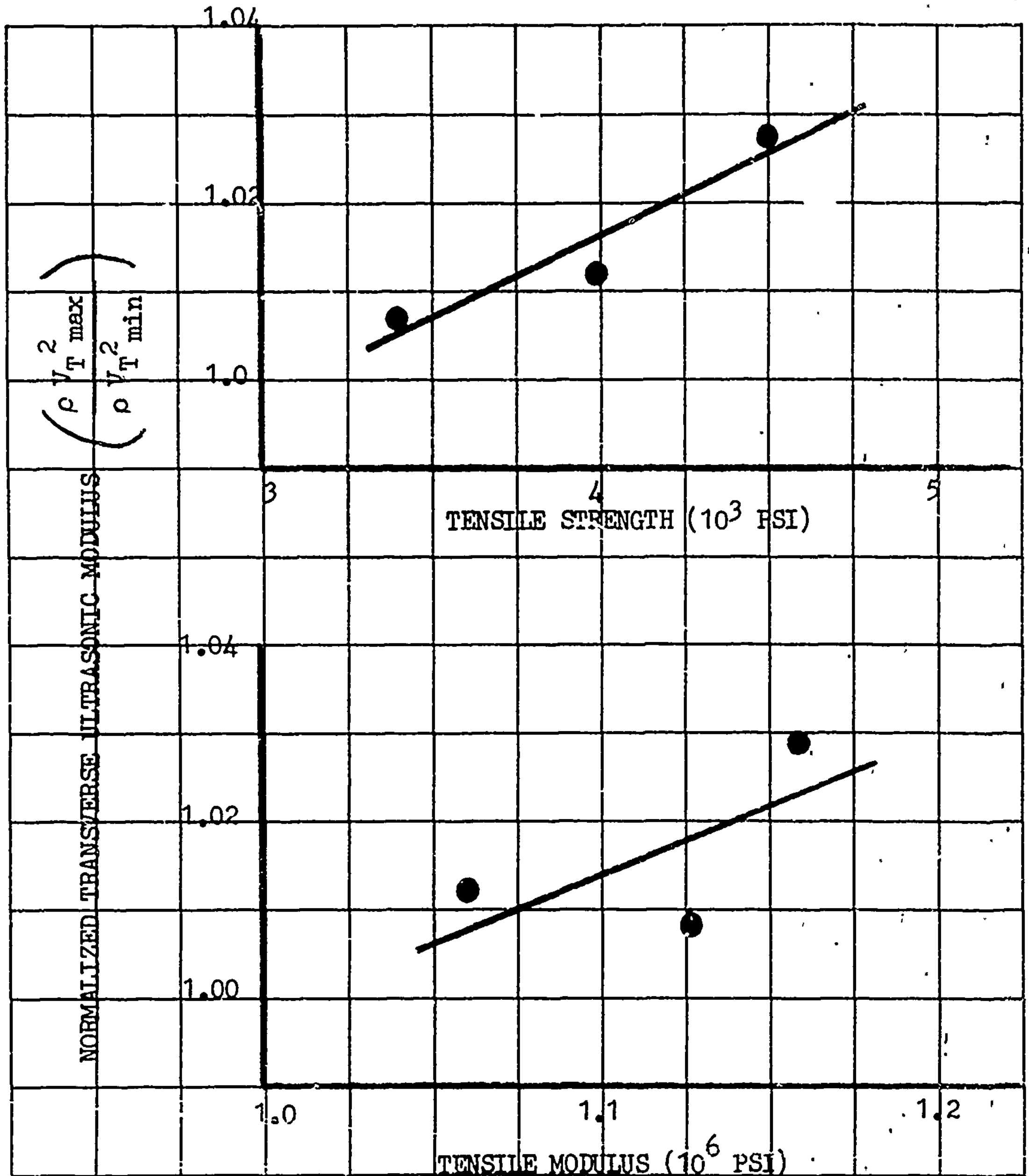

FIGURE 20 NORMALIZED TRANSVERSE ULTRASONIC MODULUS VERSUS TRANSVERSE TENSILE STRENGTH AND TRANSVERSE MODULUS (PLATE 1117-40 MODMOR II/5206)

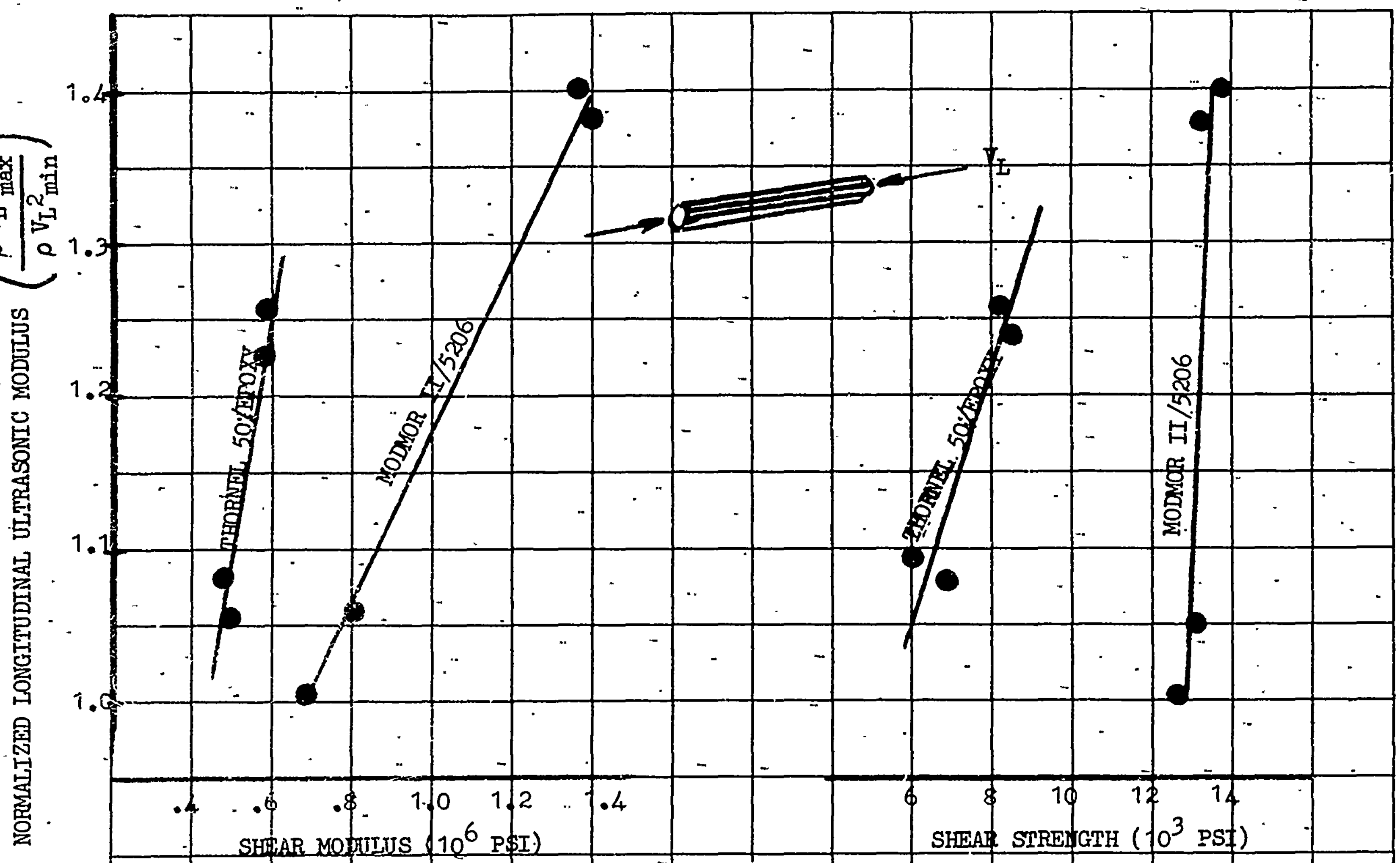

FIGURE 21 NORMALIZED LONGITUDINAL ULTRASONIC MODULUS VERSUS TORSIONAL SHEAR MODULUS AND SHEAR STRENGTH

Figure 22

Correlation of NDT and destructive test results appears to be excellent for plate 1117-37, as shown in Figure 22. Both strength and modulus exhibit meaningful trends with normalized ultrasonic modulus variations in the order of 5 to 8%. This composite was the [0/90/0/±45°] fiber orientation, and sonic velocities were measured in two perpendicular directions, i.e., the V_L direction is parallel to the 0° fibers, the V_W direction is parallel to the 90° fibers.

Figure 23

This figure presents data from two separate plates, namely 11 ... d 1117-38; and the results suggest that sonic velocity observations can ... discern the modulus values for the two different plates since we can ... more than a 25% variation in the normalized ultrasonic modulus.

Figure 24

The results obtained for Thornel 50/epoxy Plate 1117-41 appear in this illustration, and once again, an interrelation is suggested with the NDT measurements and the destructively determined modulus and strength values. The variations in NDT observables are of the order of 8%, and both the longitudinal (0°) and transverse (90°) velocities responded to the composite properties variations.

Figure 25

Ultrasonic measurements and their corresponding mechanical properties for MOD II/5206 epoxy in the [0/±45/±45/90] orientation are shown in Figure 25. The trends appear to be more significant for modulus than for strength, with observed NDT data variations of the order of 6.0%.

It should be noted that the point count nodal analysis void content determinations have not yet been completed for the MOD II/5206 composite; therefore, at this time we cannot report precise void contents and their relationships with the NDT and destructive test data.

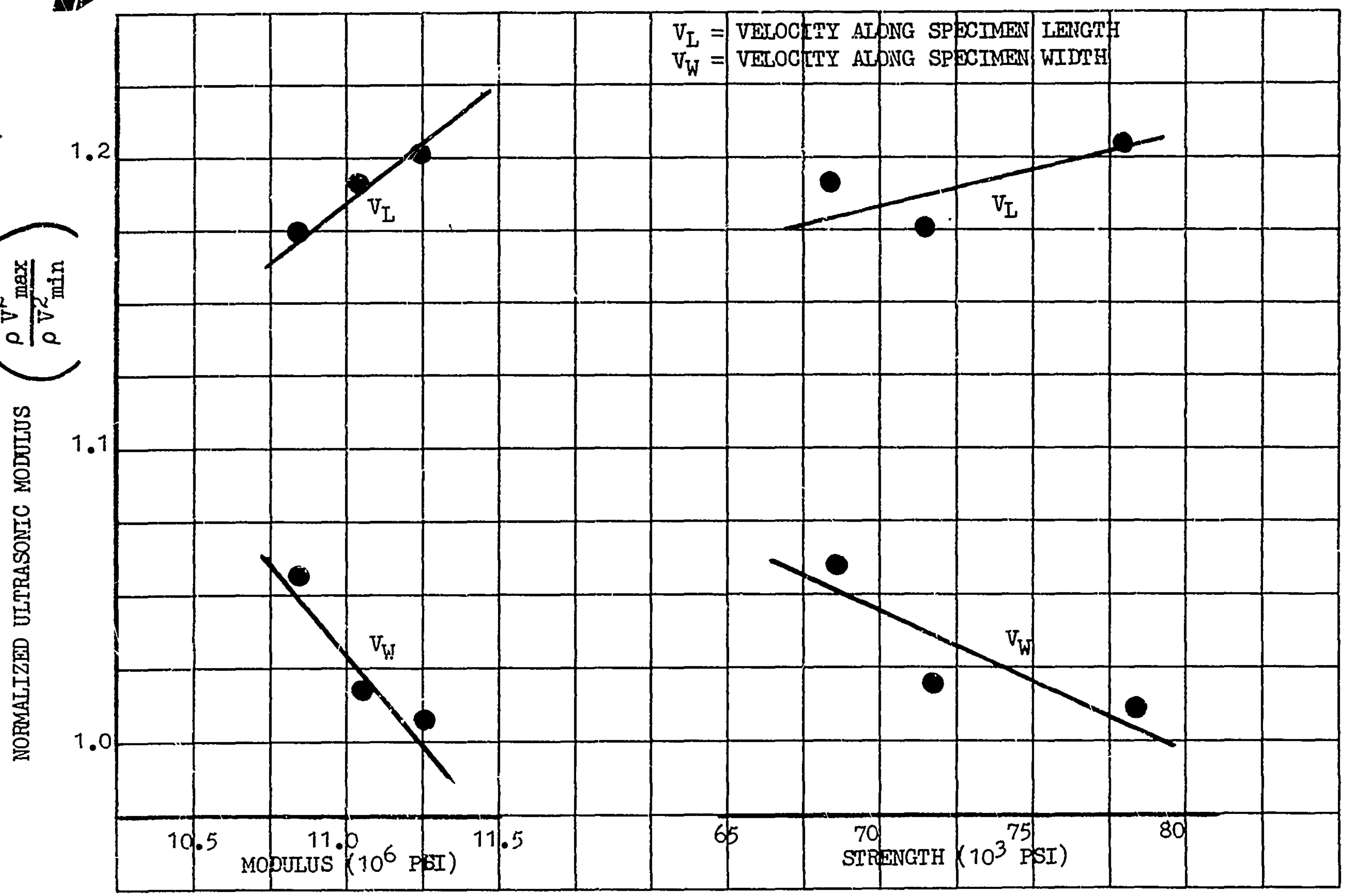

FIGURE 22 NORMALIZED ULTRASONIC MODULUS (LONGITUDINAL AND TRANSVERSE) VERSUS THE LONGITUDINAL MODULUS AND LONGITUDINAL STRENGTH (MODMOR II/5206 PLATE 1117-37, 0/90/0/±45)

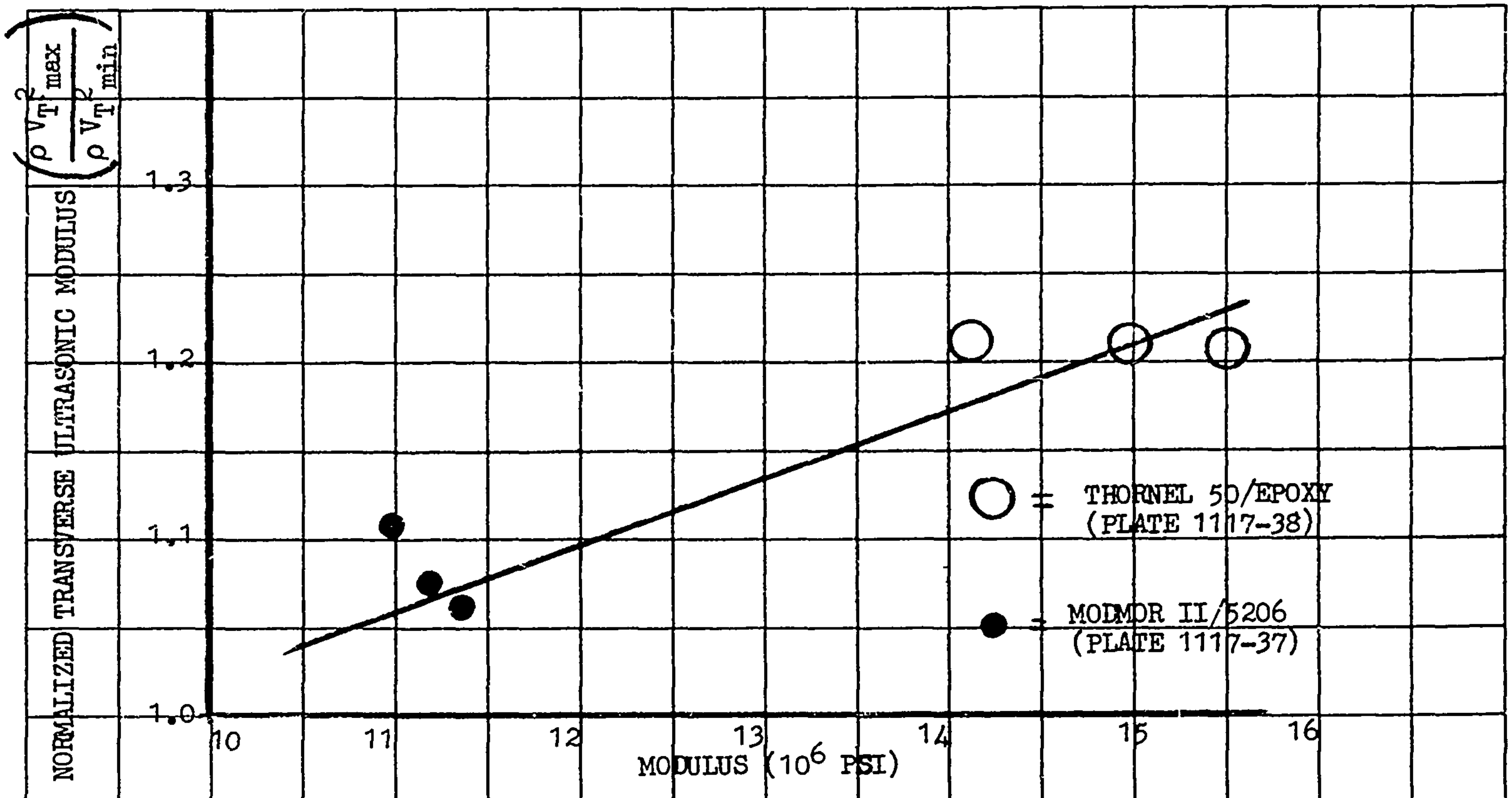

FIGURE 23 DEPENDENCE BETWEEN THE NORMALIZED TRANSVERSE ULTRASONIC MODULUS AND THE LONGITUDINAL MODULUS OF 0/90/0/+45 GRAPHITE EPOXY SPECIMEN

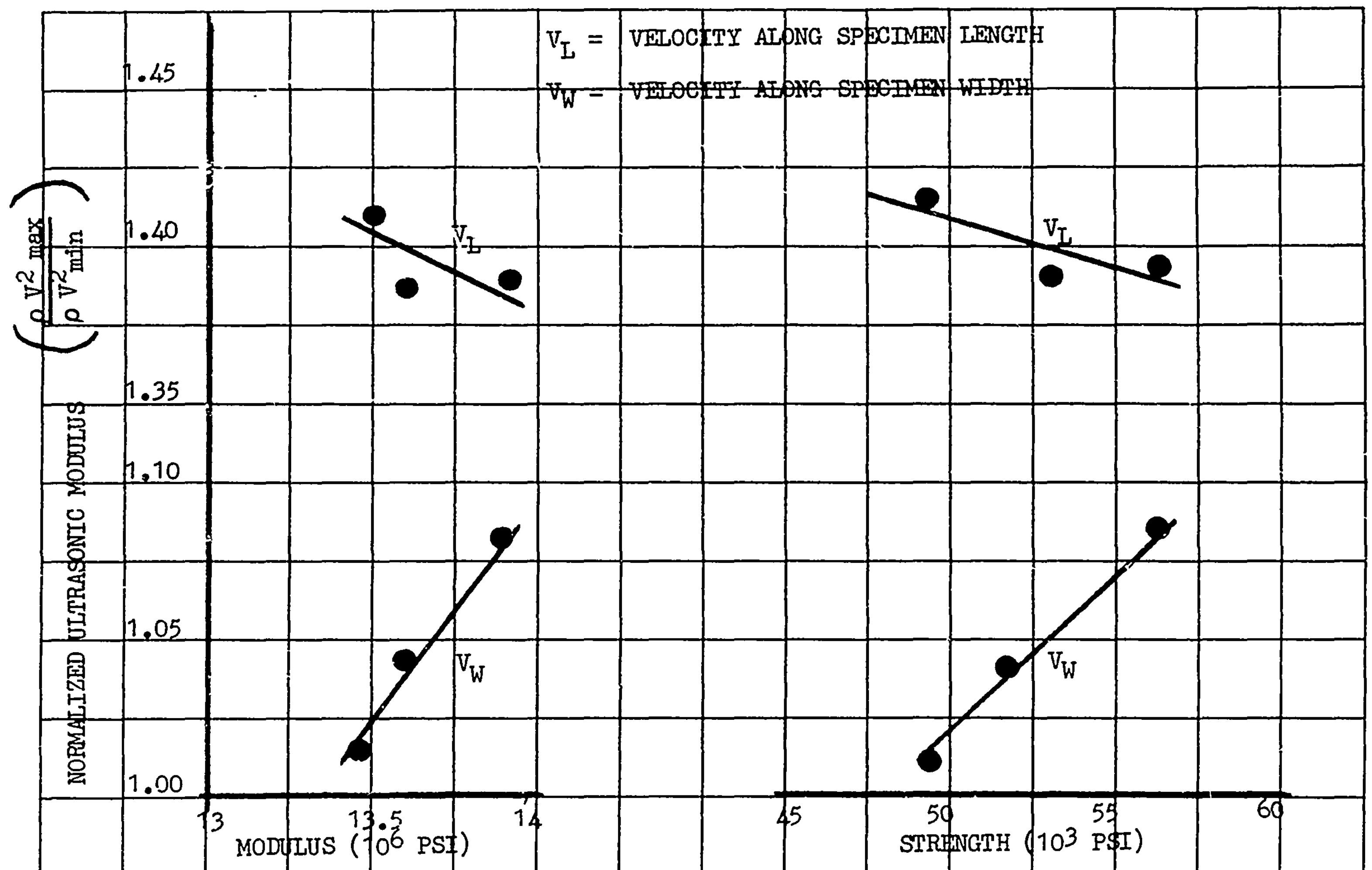

FIGURE 24 NORMALIZED ULTRASONIC MODULUS (LONGITUDINAL AND TRANSVERSE) VERSUS LONGITUDINAL MODULUS AND LONGITUDINAL STRENGTH (THORNEL 50/EPOXY, PLATE 1117-41, 0/90/0/±45)

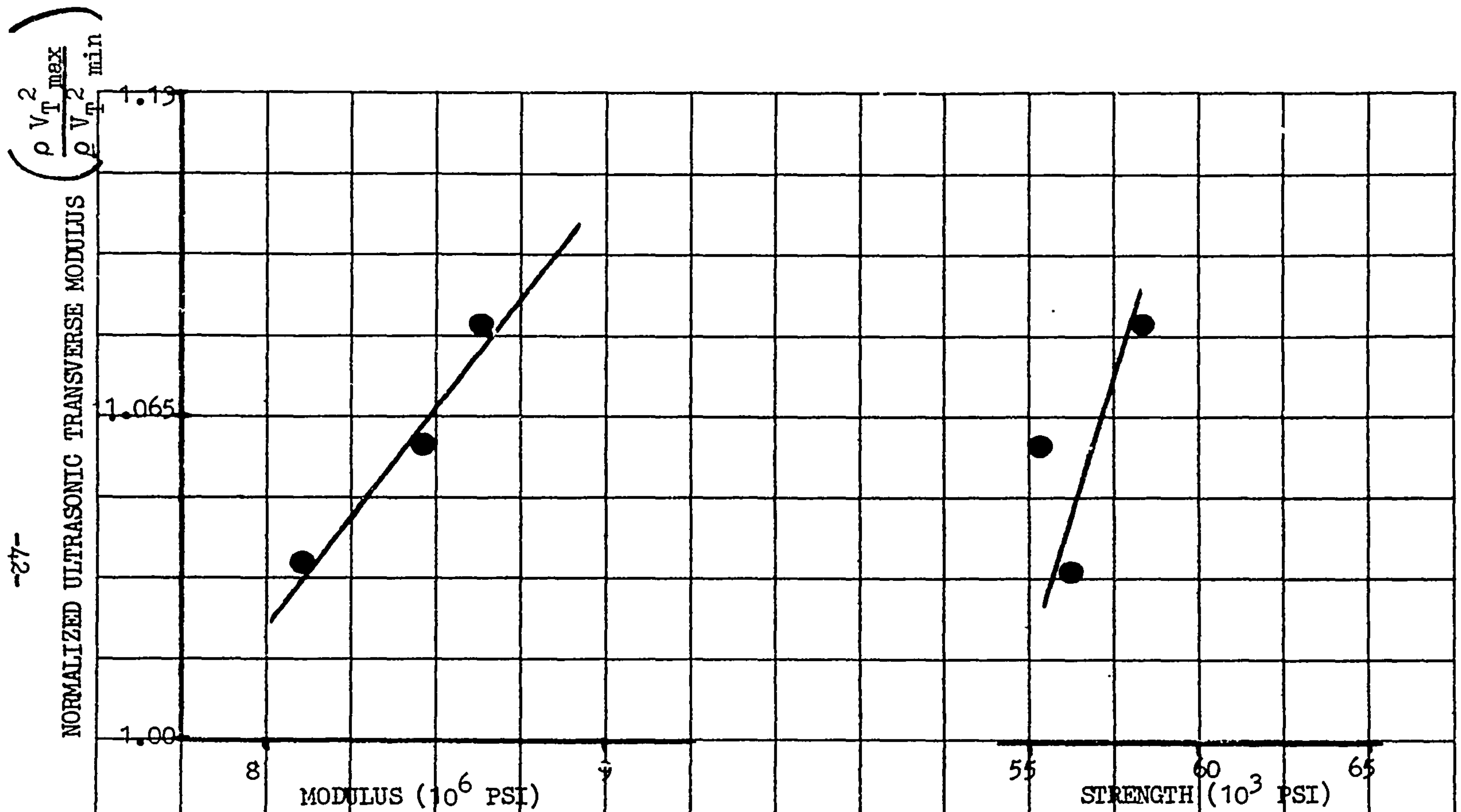

FIGURE 25 NORMALIZED ULTRASONIC TRANSVERSE MODULUS VERSUS LONGITUDINAL MODULUS AND LONGITUDINAL STRENGTH (MODMOR II/5206, 0/+45/+45/90, PLATE 1117-45

SECTION V

5.0 CONCLUSIONS AND RECOMMENDATIONS

Conclusions

On the basis of the previous observations, we can list the following conclusions:

a. Useable correlations exist between ultrasonic and destructively measured properties in both the Thornel 50 and Mod II/epoxy composites.

b. The effects of voids on the mechanical properties is summarized in Table 6. These effects are due to void contents which might be encountered in typical production procedures. Exact porosity levels, as determined by a point count nodal analysis, have not yet been determined for all of the panels; however, based upon resin burnout and acid digestion test data the void content ranges from 1 to ~ 5%. The percentage listed in Table 6 represents the change in the property between the 1% and 5% void content samples.

c. It is also worth noting that distinct differences are observed between the two composite systems. Furthermore, in some instances (transverse tension and elevated temperature tension tests on [0/90/0/±45°] Thornel 50), voids appear to increase certain mechanical properties.

d. The voids degradation effects on the unidirectional composite strength ranges from 14 to 48%.

e. Substantial strength degradation is also noted for the angle ply composite [0/90/0/±45°], with percentages of degradation varying from 16 to 27%. Thus, it is of practical significance to study such voids effects.

Recommendations

After detailed review of the past year-long effort, we arrived at the following recommendations:

a. A point count nodal analysis should be completed on the representative specimens of the materials whose properties are summarized in Table 6.

b. A point count nodal analysis should be performed on the Modmor II/5206 specimens and an effort to obtain correlations between the following should be pursued:

1) void content and NDT
2) mechanical properties and NDT
3) porosity and mechanical properties

c. A correlation should be sought relating the void content as determined by the point count nodal analysis to one of the more common laboratory procedures such as resin burnout, acid digestion, or a thermogravimetric analysis.

d. An effort should be directed toward an investigation of the effects of humidity on high and low porosity graphite epoxy composites.

TABLE 6

SUMMARY OF VOIDS EFFECTS FOR TYPICAL RANGES OF POROSITY, THORNEL 50 & MOD II/EPOXY

Material	Fiber Orientation	Test	Temperature	Average Percentage Change due to Voids Effects			
				Strength	Modulus	Failure Strain	Poisson's Ratio
Thornel 50	0°	Longitudinal Tension	75°F	14.0%	10.0%	5.0%	10.0%
		Longitudinal Tension	250°F	25.0	7.1%	16.0%	13.0%
Thornel 50	90°	Transverse Tension	75°F	32.0%*	6.3%	25.0%	12.0%
		Transverse Tension	250°F	16.6%	18.0%	14.0%	37.0%
Thornel 50	0°	Torsion Rod	75°F	26.4%	13.0%		
Thornel 50	[0/90/0/±45°]	Tension	75°F	26.6%	7.4%	12.0%	12.0%
		Tension	250°F	16.1%	13.0%	41.0%	4.0%
MOD II	0°	Longitudinal Tension	75°F	48.0%	13.0%	34.0%	10.0%
		Longitudinal Tension	250°F	39.8%	7.0%	30.0%	9.0%
MOD II	90°	Transverse Tension	75°F	36.6%	4.0%	7.5%	17.0%
		Transverse Tension	250°F	18.5%	33.0%	10.5%	7.1%
MOD II	0°	Torsion Rod	75°	14.1%	58.0%		
MOD II	[0/90/0/±45°]	Tension	75°	27.0%	11.0%	15.0%	
		Tension	250°	20.7%	11.0%	11.0%	21.0%

*These values are based on the gross voids estimates which will require further precise checking using a nodal point count metallographic analysis.

SECTION VI

6.0 REFERENCES

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